REPORT DOCUMENTATION PAGE

Form Approved OMB NO. 0704-0188

1. REPORT DATE (DD-MM-YYYY) 31-01-2017	2. REPORT TYPE Final Report	3. DATES COVERED (From - To) 15-Sep-2012 - 14-Sep-2015

4. TITLE AND SUBTITLE
Final Report: Topic 10.3 - Tailoring Grain Boundary Chemistry for Failure Resistant Nanostructured Metals

5a. CONTRACT NUMBER
W911NF-12-1-0511

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
611102

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

6. AUTHORS
Timothy J. Rupert

7. PERFORMING ORGANIZATION NAMES AND ADDRESSES
University of California - Irvine
5171 California Avenue, Suite 150

Irvine, CA 92697 -7600

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES)
U.S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSOR/MONITOR'S ACRONYM(S)
ARO

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
62262-MS.10

12. DISTRIBUTION AVAILIBILITY STATEMENT
Approved for Public Release; Distribution Unlimited

13. SUPPLEMENTARY NOTES
The views, opinions and/or findings contained in this report are those of the author(s) and should not contrued as an official Department of the Army position, policy or decision, unless so designated by other documentation.

14. ABSTRACT

This final report summarizes three years of research on the usage of grain boundary structure as a material variable able to control the toughness and ductility of nanostructured metals. Experiments on nanocrystalline Ni-W alloys were used to isolate the importance of interfacial disorder on failure, showing that additional disorder is welcome if trying to create tough materials. A strategy for amplifying this effect through the planned inclusion of amorphous grain boundary complexions was then developed, with Cu-Zr used as a model system. We have created a materials

15. SUBJECT TERMS
metals; nanostructure; grain boundary complexions; mechanical behavior; alloys

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON Timothy Rupert
a. REPORT UU	b. ABSTRACT UU	c. THIS PAGE UU	UU		19b. TELEPHONE NUMBER 949-824-4937

Standard Form 298 (Rev 8/98)
Prescribed by ANSI Std. Z39.18

Report Title

Final Report: Topic 10.3 - Tailoring Grain Boundary Chemistry for Failure Resistant Nanostructured Metals

ABSTRACT

This final report summarizes three years of research on the usage of grain boundary structure as a material variable able to control the toughness and ductility of nanostructured metals. Experiments on nanocrystalline Ni-W alloys were used to isolate the importance of interfacial disorder on failure, showing that additional disorder is welcome if trying to create tough materials. A strategy for amplifying this effect through the planned inclusion of amorphous grain boundary complexions was then developed, with Cu-Zr used as a model system. We have created a materials processing route capable of producing nanostructured metal powders with amorphous intergranular films. These powders demonstrate a concurrent increase in both ductility and strength over pure nanostructured Cu, proving that these two material properties are not mutually exclusive and going against a traditional materials design paradigm. Atomistic computer simulations were used throughout the project to provide mechanistic understanding and to guide alloy selection efforts. In addition to excellent mechanical properties, our alloys demonstrated very high thermal stability and are promising candidates for future work on bulk nanostructured materials.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received		Paper
01/31/2017	1	Amirhossein Khalajhedayati, Timothy J. Rupert. Emergence of localized plasticity and failure through shear banding during microcompression of a nanocrystalline alloy, Acta Materialia, (08 2013): 326. doi:
01/31/2017	8	Amirhossein Khalajhedayati, Zhiliang Pan, Timothy J. Rupert. Manipulating the interfacial structure of nanomaterials to achieve a unique combination of strength and ductility, Nature Communications, ( ): 10802. doi:
01/31/2017	7	Amirhossein Khalajhedayati, Timothy J. Rupert. High-Temperature Stability and Grain Boundary Complexion Formation in a Nanocrystalline Cu-Zr Alloy, JOM (Journal of The Minerals, Metals & Materials Society), ( ): 2788. doi:
01/31/2017	6	Amirhossein Khalajhedayati, Timothy J. Rupert. Disruption of Thermally-Stable Nanoscale Grain Structures by Strain Localization, Scientific Reports, ( ): 10663. doi:
01/31/2017	5	Zhiliang Pan, Timothy J. Rupert. Amorphous intergranular films as toughening structural features, Acta Materialia, ( ): 205. doi:
02/07/2017	2	Amirhossein Khalajhedayati,Timothy J. Rupert. Emergence of localized plasticity and failure through shear banding during microcompression of a nanocrystalline alloy, Acta Materialia, (02 2014): 326. doi: 10.1016/j.actamat.2013.10.074
02/07/2017	3	Timothy J. Rupert. Solid solution strengthening and softening due to collective nanocrystalline deformation physics, Scripta Materialia, (06 2014): 44. doi: 10.1016/j.scriptamat.2014.03.006
08/29/2014	4	Zhiliang Pan, Timothy J. Rupert. Damage nucleation from repeated dislocation absorption at a grain boundary, Computational Materials Science, (10 2014): 0. doi: 10.1016/j.commatsci.2014.07.008
TOTAL:		**8**

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received	Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

1. “Doping Nanocrystalline Alloys to Improve Strength and Toughness,” THERMEC International Conference on Processing and Manufacturing of Advanced Materials, May 2016, Graz, Austria.
2. “Formation and Toughening Effects of Amorphous Interfacial Phases,” International Symposium on Plasticity, January 2016, Kona, HI.
3. “Tuning Grain Boundary Structure to Control the Mechanical Behavior of Nanostructured Metallic Alloys,” Materials Research Society (MRS) Fall Meeting, December 2015, Boston, MA.
4. “Controlling Grain Boundary Structure and Properties with Segregation Engineering,” University of Florida – Department of Materials Science and Engineering, November 2015, Gainesville, FL.
5. “Nanoscale Amorphous Intergranular Films: Mechanical Properties and Interfacial Thermodynamics,” Materials Science & Technology (MS&T) Conference and Exhibition, October 2015, Columbus, OH.
6. “Plasticity and failure of nanocrystalline alloys probed with small-scale mechanical testing,” International Materials Research Congress, August 2015, Cancun, Mexico.
7. “Using amorphous complexions to tailor the mechanical behavior of nanostructured metals,” International Workshop on Interfaces, September 2015, Bear Creek, PA.
8. “Doping Nanocrystalline Metals to Improve Ductility and Toughness,” Mackenzie Presbyterian University, April 2015, São Paulo, Brazil.
9. “Atomistic Modeling of Grain Boundary Complexions: Toughening Effects and Interface Thermodynamics,” The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition, March 2015, Orlando, FL.
10. “Controlling Grain Boundary Structure and Properties with Segregation Engineering,” Boise State University – Materials Science and Engineering, March 2015, Boise, ID.
11. “Complexion Engineering in Nanostructured Materials,” Pennsylvania State University – Materials Science and Engineering, December 2014, State College, PA.
12. “Creating tough and thermally stable nanocrystalline Cu by grain boundary doping and complexion engineering,” Materials Research Society (MRS) Fall Meeting, November 2014, Boston, MA.
13. “Damage Nucleation from Dislocation-Grain Boundary Interactions: Mechanisms and Toughening Strategies,” Materials Science & Technology (MS&T) Conference and Exhibition, October 2014, Pittsburgh, PA.
14. “Catastrophic Failure of Nanocrystalline Metals: Mechanisms and Novel Toughening Strategies,” Fraunhofer Institute for Mechanics of Materials IWM, September 2014, Freiburg, Germany.
15. “Doping Nanocrystalline Alloys to Improve Strength and Toughness,” Materials Science Engineering (MSE 2014), September 2014, Darmstadt, Germany. (KEYNOTE)
16. “Tailoring Grain Boundary Structure to Control the Mechanical Behavior of Nanocrystalline Alloys,” The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition, February 2014, San Diego, CA.
17. “The Effects of Grain Boundary Volume Fraction and Relaxation State on Uniaxial Plasticity of Nanocrystalline Metals,” The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition, February 2014, San Diego, CA.
18. “Novel Solid Solution Effects on the Strength of Nanocrystalline Metals,” UCI-UNIST Engineering Workshop, February 2014, Irvine, CA.
19. “The Influence of Grain Boundary Structure on Plastic Flow and Failure in Nanocrystalline Alloys,” THERMEC International Conference on Processing and Manufacturing of Advanced Materials, December 2013, Las Vegas, NV.
20. “Catastrophic Shear Banding in Nanocrystalline Metals and the Importance of Grain Boundary Structure,” University of California, Riverside – Materials Science and Engineering, November 2013, Riverside, CA
21. “Uniaxial Flow and Failure of Nanocrystalline Alloys Investigated by Focused Ion Beam Microscopy,” Southern California Society for Microscopy and Microanalysis Spring Symposium, March 2013, Los Angeles, CA.
22. “The Influence of Atomic Grain Boundary Structure on Plastic Flow in Nanocrystalline Alloys,” Materials Research Society (MRS) Fall Meeting, November 2012, Boston, MA. (poster)
23. “Grain Boundary Structure and Chemistry: Impact on Nanocrystalline Plasticity,” Society of Engineering Science (SES) Annual Technical Meeting, October 2012, Atlanta, GA.

Number of Presentations: 23.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received	Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received	Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received	Paper

TOTAL:

Number of Manuscripts:

Books

Received	Book

TOTAL:

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

1. TMS Structural Materials Division JOM Best Paper Award (2016) - T. Rupert
2. UCI Engineering, Early Career Award for Faculty Excellence in Research (2016) - T. Rupert
3. Army Research Office, Young Investigator Program (YIP) Award (2016) - T. Rupert
4. Scripta Materialia, Outstanding Reviewer Award (2015) - T. Rupert
5. Department of Energy, Early Career Research Program Award (2015) - T. Rupert
6. TMS Young Leader Professional Development Award (2015) - T. Rupert
7. Hellman Faculty Fellow (2014) - T. Rupert
8. MRS Science as Art, 2nd Place (2014) - A. Khalajhedayati
9. NSF Faculty Early Career Development (CAREER) Award (2013) - T. Rupert

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
Amirhossein Khalajhedayati	1.00	
Simon Pun	1.00	
FTE Equivalent:	**2.00**	
Total Number:	**2**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
Zhiliang Pan	0.33
FTE Equivalent:	**0.33**
Total Number:	**1**

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
Timothy J. Rupert	0.08	
FTE Equivalent:	**0.08**	
Total Number:	**1**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED	Discipline
Daniel Grant	1.00	Mechanical Engineering
Trent Nash	0.25	Mechanical Engineering
Simon Pun	1.00	Materials Science and Engineering
Carlos Ramirez	0.25	Mechanical Engineering
FTE Equivalent:	**2.50**	
Total Number:	**4**	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 4.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 4.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 3.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 3.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:...... 0.00

Names of Personnel receiving masters degrees

NAME	
Simon Pun	
Total Number:	1

Names of personnel receiving PHDs

NAME	
Amirhossein Khalajhedayati	
Total Number:	1

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

This project sought to understand how the failure resistance of nanocrystalline metals can be tuned by controlling grain boundary structure. Nanocrystalline metals have exceptionally high strength, but limited ductility and toughness. The recent identification of grain boundary "complexions" introduces a new materials design variable that should be very powerful for nanocrystalline materials: atomic grain boundary structure. We hypothesized that premature failure in nanocrystalline metals can be delayed by creating nanocrystalline alloys that contain amorphous intergranular films (AIFs), which can act as efficient sinks for the dislocation mechanisms that control plasticity in grain sizes from 15 to 100 nm.

Specific goals for this project included: (i) development of a robust technique for measuring flow and failure in small volumes of nanocrystalline materials, (ii) understanding the importance of atomic grain boundary structure on failure, (iii) identification of processing routes for creating nanostructured alloys with a variety of grain boundary structures through intelligent doping, and (iv) production of tough nanocrystalline powders suitable for scale-up to bulk nanocrystalline materials.

The project included three complementary task lines:
1) Production of Nanostructured Metals with Different Grain Boundary States
2) Characterization of Microstructures and Interfacial States
3) Quantification of Uniaxial Flow and Failure

This document is intended as a summary of the achievements and progress during this three year research project. For each task line, experimental and theoretical advances are highlighted below.

Task Line 1: Production of Nanostructured Metals with Different Grain Boundary States

This task line involved the creation of nanocrystalline metals with a variety of grain boundary states. Experimental tools including electrodeposition, mechanical alloying, and drop quenching were used to fabricate different nanocrystalline alloys and alter their boundary structure. Theoretical tools such as Monte Carlo and molecular dynamics simulations were brought to bear on this problem, to give added power to our process optimization.

1. We used pulsed electrodeposition to create nanocrystalline Ni-W films. This system can be used to create nanocrystalline metals with average grain sizes from 3-100 nm, meaning the effect of grain size on failure can be isolated. Three nanocrystalline Ni-W samples were studied with microcompression, probing failure when different deformation mechanisms control nanocrystalline plasticity. These alloys also have nonequilibrium grain boundaries in their as-deposited state which can be relaxed with annealing. By relaxing grain boundary structure and comparing to as-deposited behavior, we were able to perform proof-of-concept experiments to show the importance on grain boundary structure on failure. The results of these experiments will be described in more detail under Task Line 3 below.

2. Cu-Zr was chosen as the model system through which we could explore how grain boundary structure and toughness can be controlled with doping. Zr should strongly segregate to the grain boundaries (negligible solubility in the Cu lattice) and Cu-Zr is an excellent binary glass forming system (negative heat of mixing, large atomic size mismatch), making this alloy an excellent candidate for complexion engineering. Preliminary ball milling experiments in our lab showed that a minimum grain size of ~30 nm was common for Cu-based alloys created with a SPEX mill. The ball mill is inside of a glove box under an Ar atmosphere to avoid oxidation during milling. During preliminary milling experiments, we explored the effects of powder purity, ball-to-powder ratio, amount of stearic acid (process control agent), and milling time. Powder contamination proved to be an important concern. Our early powders contained unmixed Zr particles, as well as impurities such as ZrH and ZrC. By increasing the ball-to-powder ratio to 10 and reducing the stearic acid addition to 1%, we were able to completely remove the Zr and ZrH. The reduction in process control agent meant that longer milling times had to be used. Some small amount of ZrC is still occasionally observed and other process control agents such as graphite should be explored to eliminate this issue.

3. A procedure was developed for annealing powder samples at high temperatures and then quickly quenching to freeze in any complexion structures that are formed. The Cu-Zr powder samples were encapsulated in quartz tubes under vacuum, to avoid oxidation during annealing. A drop quench furnace was used consisting of a vertical tube furnace situated over a 30 gallon water reservoir. Each powder-filled tube was hung in the furnace to anneal, and then the wire holding the tube was cut so that it fell quickly into the quenching liquid. We mainly explored annealing at 950 °C, a temperature very close to the solidus temperature, since such a treatment would be most likely to create a thick amorphous film at the grain boundaries. However, additional annealing experiments were run at 550 °C, 750 °C, and 850 °C, to explore the temperature dependence of complexion formation. To isolate the effects of AIFs from other variables which change during annealing such as grain size and internal strain, additional powder samples were annealed but then slowly cooled to room temperature. Since AIFs are only thermodynamically stable at high temperatures, no amorphous complexions are expected in this case. Characterization results are presented under Task Line 2.

4. To guide choices concerning alloy composition and annealing, we used a hybrid molecular dynamics/Monte Carlo (MD/MC) technique to explore how composition and temperature affect grain boundary structure. Such a technique allows for structural relaxation, thermal vibrations, and the exchange of atom types at the same time. This study allowed us to predict different

structural states that are accessible in the Cu-Zr system and find the conditions most likely to achieve these experimentally, as well as understand how these structures depend on variables such as grain boundary character. We were also able to understand how complexion and intermetallic formation compete.

Task Line 2: Characterization of Microstructures and Interfacial States

Experimental tools including x-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) were used to characterize the different nanocrystalline alloys produced in Task Line 1. A focused ion beam (FIB) microscope was used to create the site-specific TEM samples.

1. The nanocrystalline Ni-W samples that were used in the proof-of-concept experiments were annealed at 300 °C to relax the grain boundaries, and TEM characterization was used to show that no grain growth occurred and that no second phase was precipitated. This characterization confirmed that only subtle relaxation of the grain boundary structure could be occurring, isolating grain boundary state as a structural variable.

2. During the initial development of our microcompression testing on nanocrystalline Ni-W, we observed that strain localization in shear bands is a larger problem than previously thought. We performed site-specific TEM investigation of shear bands from nanoindentation and microcompression. Mechanically-induced grain growth was found inside of the shear bands, with the thickness of the coarsened region a function of the plastic strain in the band. Nanocrystalline Ni-W has been shown to have excellent thermal stability and does not coarsen during homogeneous plastic deformation. However, strain localization can overcome this barrier and drive structural evolution which aids catastrophic failure. This finding demonstrates that even dopant-stabilized nanostructures can become dynamic during shear banding, highlighting an important failure mechanism for nanocrystalline alloys.

3. The Cu and Cu-Zr powders were characterized in their as-milled state, to first allow for process optimization and then to give baseline data before annealing. We found that while grain sized reached its minimum value of ~30 nm after only 12 hours of milling, unmixed Zr particles remained in the powder. By increasing the milling time, ball-to-powder ratio, and stearic acid content as described in Task Line 1, fully mixed powders were achieved. XRD and TEM show that the latest as-milled powders contain only a solid solution FCC phase where Zr replaces Cu on its lattice.

4. TEM has been used to extensively characterize the powder samples after annealing and quenching. The average grain size increased to ~45 nm after annealing for 1 hour at 950 °C, meaning the material is still nanocrystalline and rampant grain growth is not a problem even at these high temperatures. High resolution TEM was used to investigate local grain boundary structure in annealed Cu-Zr powders which were either slowly cooled in air (not conducive to AIF formation) or drop quenched (likely to form AIFs). We were able to obtain samples with and without amorphous intergranular films and these two samples give us specimens that can be mechanically tested to isolate the effect of disordered complexions, since all other structural variables are the same. Further details will be given in Task Line 3.

5. TEM was also used to study the details of grain boundary chemistry. High-angle annular dark-field scanning TEM and energy dispersive spectrometry confirm that grain boundary regions have elevated Zr concentrations, while the crystal interiors are pure Cu. This is important because the expectation of grain boundary segregation was one of the reasons for choosing Zr as our dopant.

Task Line 3: Quantification of Uniaxial Flow and Failure

This task line involved quantitative mechanical testing of plastic flow and failure of the nanocrystalline metals created in Task Line 1. Experimental tools including FIB, nanoindentation, and microcompression were used to test nanocrystalline alloys with different boundary structures. Theoretical tools such as molecular dynamics simulations were also brought to bear on this problem, to help interpret experimental trends and guide future alloy design.

1. A reliable, repeatable procedure for measuring the plastic flow and failure properties of nanocrystalline metals was developed. We use a nanoindenter with a flat punch tip to compress micropillars created with the FIB. While this technique has been used almost exclusively in the past to measure the effects of external sample size on mechanical behavior, we instead use pillars that are much larger than our internal microstructure to avoid any size effect and probe the bulk-properties of the material. Taper-free pillars with limited Ga damage are created using a lathe milling technique. This method can be used to test perform multiple experiments inside of a single powder particle, allowing for rapid measurement of mechanical properties and fast alloy development. Since a relatively small volume of material is used, microcompression also removes complications from processing defects such as voids and hydrogen pitting. We have shown quite clearly that uniaxial experiments such as these are essential for probing mechanical behavior. Although hardness is often used as a metric of strength, we observe that

it is in fact not proportional to yield strength in specimens which do not experience measureable plastic strain.

2. Microcompression was used to probe the uniaxial deformation of samples with grain sizes of 5, 15, and 90 nm, providing baseline data and an understanding of how different nanocrystalline grain sizes fail. To the best of our knowledge, this is the first systematic study of uniaxial failure over the entire Hall-Petch breakdown. The largest grain size of 90 nm could be compressed to more than 25% strain without failing, and deformation remained homogeneous. The 15 nm grain size experienced pronounced strain softening after yield and failed suddenly through shear banding at applied strains of 10-20%. The finest grain size of 5 nm showed elastic-perfectly plastic deformation with no apparent strain hardening or softening after yield. After plastic strains of only a few percent, strain localization occurs and these specimens fail through shear banding that resembles the behavior of metallic glasses. These results highlight the fact that strain localization failure is a significant problem for very fine nanocrystalline metals. We also performed a study of the mechanical behavior of the Ni-W samples after their nonequilibrium boundary structures were relaxed with low temperature heat treatments. This served as a proof-of-concept experiment to understand how increasing the order of interfacial structure affects failure. Relaxation of nonequilibrium grain structure strengthens nanocrystalline metals, but makes our two finest grain sizes much more susceptible to strain localization. For the 15 nm grain size, the strain-to-failure is reduced to 5-8%, while the 5 nm grain size sample shows no appreciable plastic strain before shear banding causes failure. These results prove the overarching concept of this study, that grain boundary state can dramatically affect nanocrystalline failure. Grain boundary disorder is necessary for stable plasticity at small grain sizes, motivating our goal of adding a stable amorphous grain boundary phase.

3. Microcompression was used to explore the uniaxial flow and failure of our Cu and Cu-Zr alloys. Pure Cu has a strength of ~800 MPa, which is comparable to literature reports for nanocrystalline Cu, but also demonstrates strong strain softening. Cu-Zr with clean grain boundaries is stronger, even though the grain size has increase to ~45 nm. Zr segregation to the grain boundaries should decrease the grain boundary energy and will increase dislocation pinning as it moves through a single grain. Both pure Cu and Cu-Zr with clean grain boundaries experience brittle failure and cracking of the pillar. Since compression is used, the sample can still sustain a load unlike a tension experiment. Alternatively, Cu-Zr with AIFs demonstrated uniform compression and homogeneous plasticity. No cracking of the pillars was found, providing evidence that the addition of AIFs serves to toughen the material.

4. Although our microcompression results suggested that AIFs have a positive effect on nanocrystalline toughness, compression is a constrained testing mode and failure is difficult to measure with such experiments. Additional pillars were therefore bent in situ inside of the SEM with an Omniprobe actuator, to directly observe failure. Pure Cu was extremely brittle, with the pillars cracking near the base with no appreciable plastic strain. Cu-Zr with clean grain boundaries experienced some plastic strain but still failed and started to fracture apart near the base. Again, Cu-Zr with AIFs out-performed all other samples. The pillar could be bent to high angles without developing full cracks. Instead, extensive plastic flow was observed and the pillars retained their structural integrity. Both microcompression and in situ bending show a toughening effect, proving that AIFs can be advantageous and delay premature failure of nanocrystalline metals. By estimating the plastic strain-to-failure from the various pillars, we found an order of magnitude increase in the ductility can be obtained by adding AIFs. Pure nanocrystalline Cu failed at 5% strain while the Cu-Zr with AIFs failed at strain >50%, an outstanding results for a nanostructured metal.

5. To provide a physical understanding for our observations, MD simulations have been used to simulate dislocation-grain boundary interactions and study crack nucleation. The absorption of a dislocation at nanocrystalline grain boundaries can lead to local stress concentrations, which we hypothesize can then develop into crack damage after repeated absorption. We developed a simulation methodology where a virtual dislocation source is operated as the cell is sheared. Local stresses are only high immediately near the source and crack nucleation can be studied as dislocations are absorbed. A uniaxial strain applied to the cell provides a driving force for crack nucleation. A clean grain boundary was first studied. We found that atomic shuffling events determine how the free volume brought by the incoming dislocation is accommodated and multiple thermodynamically equivalent starting configurations are required to quantify the damage resistance of a given grain boundary. After studying a clean boundary, AIFs with different thicknesses were added and the simulations repeated. The two interfaces were manually doped and melted, then quenched to freeze in the amorphous structure. The AIF delays the nucleation event to substantially larger strains or number of dislocations absorbed. A monotonic increase in grain boundary damage resistance was found with increasing AIF thickness. A simple model based on free-volume accommodation and displacement events in the amorphous boundary was found to describe our observed trend. We also explored the damage resistance of our interface structures created by MD/MC modeling, finding similar results.

Technology Transfer

The Invention Transfer Group (ITG) at UCI Applied Innovation has decided to pursue a patent for our processing route for the creation of nanostructured powders with increased toughness and thermal stability. A “Record of Invention” disclosure has been filed and the ITG is in the process of filing a provisional patent application. Once this is accomplished, we plan on either licensing the technology or creating our own start-up company to pursue commercialization.