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RPPR Final Report
as of 22-Jun-2020

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Major Goals: This project sought to develop bulk nanostructured materials with high strength and ductility, utilizing amorphous intergranular films (AIFs) as features that enable both consolidation and enhanced mechanical properties. These amorphous films are in equilibrium, meaning they are stable grain boundary structures, but should also improve grain boundary diffusivity and the ability of an interface to absorb dislocations without cracking. The proposed research answered fundamental questions about the importance of grain boundary complexions for mechanical damage and thermal stability. What alloy compositions and heat treatment temperatures lead to the formation of different complexions in a nanocrystalline metal? How do complexions affect mechanical behavior and consolidation? How does processing affect complexion type and thickness? In the end, we were able to extend the concept of interfacial engineering, by adding another design variable to the toolbox that materials engineers can use to control mechanical properties and thermal stability simultaneously.

Specific goals for this project included:
(i) Understanding the importance of amorphous complexions on consolidation and deformation.
(ii) Measurement of mechanical properties and thermal stability.
(iii) Production of tough nanocrystalline materials in bulk form.

All of the goals above were accomplished, with more details included below.

Accomplishments: We have accomplished all of the major goals set out in our proposal. First and foremost, we have created fully dense nanocrystalline materials with a small grain size, beating all other reports in the literature. Scientifically, it is essential to recognize that the improved densification we observed was directly connected to the formation of AIFs in the samples. We also have shown definitive proof that complexion transitions can alter the tensile plasticity and failure mode of nanocrystalline metals. Finally, a number of important results were found concerning the effect of impurities on stability and mechanical behavior, how laser shock could be used to alter the glassy structure of the amorphous films, and the extension of complexions to dislocation defects.

Task Line 1: Consolidation of Powders into Bulk Nanostructured Metal Pieces
We first showed that AIFs can stabilize grain size by studying grain growth and grain boundary structure in nanocrystalline Ni-W. An alloy that was more stable at higher temperatures (without any bulk phase transformation) due to AIF formation was found, proving our initial hypothesis. These results were published in Scripta Materialia. Consolidation experiments were then performed using a simple hot pressing furnace. Powders were ball milled in a glove box filled with Ar, then cold pressed at ~25 MPa to create a green body, followed by hot pressing at 50 MPa to create bulk samples with ½” diameter. All specimens remained nanocrystalline, and a noticeable jump in density

was found that matches both experimental and theoretical predictions for AIF formation temperature. We were able to attain >99% density while retaining a nanocrystalline grain size. It is important to emphasize that the equipment and techniques used here are very common, with the samples simply heated and compressed, yet attain notable density-grain size combinations. These results were published in Advanced Engineering Materials.

Task Line 2: Robust Measurement of Local Strength and Ductility
We first used microcompression testing to measure the strength of nanocrystalline Al-Mg alloys designed to experience grain boundary segregation. These alloys had notably high strengths due to the boundary state and these results were published in Materials Science and Engineering A. We also developed uniaxial tension testing techniques to directly measure the mechanical behavior of our alloys. While developing this technique, we perfected our methods on a nanostructured high entropy alloy as part of a collaborative effort, which was published in Science Advances. These methods were then used on nanocrystalline Cu-Zr with and without AIFs, so isolate the effect of grain boundary structure. The sample with AIFs showed evidence of extensive plastic flow, with ample necking in the gauge section. The cross-section was reduced by a great deal due to the local plastic strain. In contrast, the sample with ordered grain boundaries experienced catastrophic failure with little plastic strain, which is indicative of an extremely brittle material. A manuscript describing these results is in the final stages of internal review and will be submitted shortly.

Task Line 3: Competition between Planned Metal Dopants and Unintentional Impurities
We were able to uncover the effect of various impurities with first-principles calculations, as well as understand how these impurities interact with the planned metallic dopants that are added to stabilize the grain structure. A key finding was that a combination of a larger substitutional dopant and an interstitial impurity can fill the excess free volume more efficiently and further reduce the grain boundary energy. We also found that the strengthening effects of dopants and impurities are dominated by the electronic interactions, with correct choices able to overcome any embrittlement from impurities. This work was published during the reporting period as two separate papers in Acta Materialia.

Task Line 4: Manipulation of Amorphous Complexion Structure and Properties
We collaborated with Prof. Dan Gianola at UCSB to see if the local structure and properties of AIFs can be perturbed. Using a femtosecond laser, a stress wave was sent through near-surface regions of our samples, effectively changing the free volume of the amorphous region. We found that the application of this laser leads to a dramatic reduction in hardness of our material, but this effect is reversible with low temperature annealing. This finding is scientifically very interesting, as it means the structural disorder nature of the boundaries can be tuned, and has been published in Acta Materialia. In addition, separate molecular dynamics modeling work was performed to understand how strength was connected to atomic details of complexion structure, by isolating fundamental deformation mechanisms, leading to another publication in Acta Materialia.

Task Line 5: Extension of the Complexion Concept to Dislocations
Motivated by the fact that other defects will have the local stress and structural variations that induce grain bound complexion transitions, we performed modeling work to understand linear complexions near dislocations in FCC metals. We found at last three new types of linear complexions, depending on the alloy system that was chosen. We made linear complexion diagrams that show the local compositions that are needed for a complexion transition at a given temperature, which can guide future experimental validation efforts. This work has been published in Acta Materialia.

Training Opportunities: The project supported the effort of two graduate students, including a Ph.D. student focused on alloy design, powder processing, and thermal stability as well as an M.S. student who developed in situ mechanical testing methods that were applied the alloys of interest. Two postdoctoral scholars performed atomistic simulations and consolidation experiments for the project. The postdocs were primarily supported by discretionary funds of the PI, but did contribute valuable work. All trainees were mentored in the fields of Materials Science and Solid Mechanics, giving them multi-disciplinary skills that will help them in the workplace. Each met weekly with the PI to discuss research goals and career ambitions, while also presenting in front of the research group quarterly to develop presentation skills.

Results Dissemination: The research results from this project have been distributed to the community in the form of invited talks and contributed presentations, as well as well as journal articles. Thirty-two invited talks and fourteen contributed presentations were given, spread between conferences, universities, and national laboratories. These presentations are listed in an uploaded document. Ten journal articles and one conference proceeding paper have been published to date on this project, including works appearing in top material science and engineering journals such as Acta Materialia, Advanced Engineering Materials, and Science Advances. In addition, four additional papers are either already under external peer review or in the final stages of internal review. The work published to date appears in the Products section of this report as well as the uploaded document. Copies of all journal articles resulting from this work have been uploaded to the arXiv.org repository, with PDF copies of the final articles also posted on the PI’s website.

Honors and Awards: - AIME-TMS Rossiter W. Raymond Memorial Award (2020)
- Invited Speaker, Gordon Research Conference – Physical Metallurgy (2019)
- Finalist, Robert W. Cahn Best Paper Prize for the Journal of Materials Science (March 2019)
- ASM International Bradley Stoughton Award for Young Teachers (2017)
- National Academy of Engineering U.S. Frontiers of Engineering Symposium Participant (2017)

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Technology Transfer: Nothing to Report

PARTICIPANTS:

Participant Type: PD/PI
Participant: Timothy John Rupert
Person Months Worked: 3.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Postdoctoral (scholar, fellow or other postdoctoral position)
Participant: Olivia Donaldson
Person Months Worked: 6.00 **Funding Support:**
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Other Collaborators:

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Participant: Vladyslav Turlo
Person Months Worked: 3.00 **Funding Support:**
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International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Charlette Grigorian
Person Months Worked: 9.00 **Funding Support:**
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International Collaboration:

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Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Jenna Wardini
Person Months Worked: 6.00 **Funding Support:**
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ARTICLES:

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Volume: 696 Issue: First Page #: 400
Date Submitted: 8/31/17 12:00AM Date Published: 6/1/17 7:00AM
Publication Location:
Article Title: Nanocrystalline Al-Mg with extreme strength due to grain boundary doping
Authors: Simon C. Pun, Wenbo Wang, Amirhossein Khalajhedayati, Jennifer D. Schuler, Jason R. Trelewicz, Tim
Keywords: Grain boundary strengthening; Grain boundary doping; Nanocrystalline metals; Nanoindentation; Mechanical behavior
Abstract: Nanocrystalline Al-Mg alloys are used to isolate the effect of grain boundary doping on the strength of nanostructured metals. Mg is added during mechanical milling, followed by low homologous temperature annealing treatments to induce segregation without grain growth. Nanocrystalline Al -7 at.% Mg that is annealed for 1 h at 200 °C is the strongest alloy fabricated, with a hardness of 4.56 GPa or approximately three times that of pure nanocrystalline Al. Micropillar compression experiments indicate a yield strength of 865 MPa and a specific strength of 329 kN*m/kg, making this one of the strongest lightweight metals reported to date.
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Publication Location:
Article Title: Uncovering the influence of common nonmetallic impurities on the stability and strength of a ?5 (310) grain boundary in Cu
Authors: Zhifeng Huang, Fei Chen, Qiang Shen, Lianmeng Zhang, Timothy J. Rupert
Keywords: Grain boundaries; Impurity segregation; Thermodynamic stability; Embrittlement; First-principles calculations
Abstract: Impurities are often driven to segregate to grain boundaries, which can significantly alter a material's thermal stability and mechanical behavior. To provide a comprehensive picture of this issue, the influence of a wide variety of common nonmetallic impurities (H, B, C, N, O, Si, P and S) incorporated during service or materials processing are studied using first-principles simulations, with a focus on identifying changes to the energetics and mechanical strength of a Cu ?5 (310) grain boundary. Changes to the grain boundary energy are found to be closely correlated with the covalent radii of the impurities and the volumetric deformations of polyhedra at the interface. The strengthening energies of each impurity are evaluated as a function of covalent radius and electronegativity, followed by first-principles-based tensile tests on selected impurities. The strengthening of a B-doped grain boundary comes from an enhancement of the charge density among the adjacent Cu atoms...
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Article Title: Grain boundary complexions and the strength of nanocrystalline metals: Dislocation emission and propagation
Authors: Vladyslav Turlo, Timothy J. Rupert
Keywords: Dislocations; grain boundary complexions; amorphous intergranular film; molecular dynamics simulation
Abstract: Grain boundary complexions have been observed to affect the mechanical behavior of nanocrystalline metals, improving both strength and ductility. While an explanation for the improved ductility exists, the observed effect on strength remains unexplained. In this work, we use atomistic simulations to explore the influence of ordered and disordered complexions on two deformation mechanisms which are essential for nanocrystalline plasticity, namely dislocation emission and propagation. Both ordered and disordered grain boundary complexions in Cu-Zr are characterized by excess free volume and promote dislocation emission by reducing the critical emission stress. Alternatively, these complexions are characterized by strong dislocation pinning regions that increase the flow stress required for dislocation propagation. Such pinning regions are caused by ledges and solute atoms at the grain-complexion interfaces and may be dependent on the complexion state as well as the atomic size...
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Journal: Scripta Materialia
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Date Submitted: 8/29/18 12:00AM Date Published: 9/1/18 7:00AM
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Article Title: Amorphous complexions enable a new region of high temperature stability in nanocrystalline Ni-W
Authors: Jennifer D. Schuler, Olivia K. Donaldson, Timothy J. Rupert
Keywords: complexion; nanocrystalline metal; grain growth; grain boundary segregation; interface structure
Abstract: Solute segregation is used to limit grain growth in nanocrystalline metals, but this stabilization often breaks down at high temperatures. Amorphous intergranular films can form in certain alloys at sufficiently high temperatures, providing a possible alternative route to lower grain boundary energy and therefore limit grain growth. In this study, nanocrystalline Ni-W that is annealed at temperatures of 1000 °C and above, then rapidly quenched, is found to contain amorphous intergranular films. These complexions lead to a new, unexpected region of nanocrystalline stability at elevated temperatures.
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Article Title: Femtosecond laser rejuvenation of nanocrystalline metals
Authors: Glenn H. Balbus, McLean P. Echlin, Charlette M. Grigorian, Timothy J. Rupert, Tresa M. Pollock, Daniel
Keywords: Nanocrystalline; Femtosecond lasers; Grain boundaries; Rejuvenation
Abstract: Nanocrystalline metals are distinct from traditional engineering materials due to their high concentration of grain boundaries and corresponding structural disorder at grain boundaries. The effect of local disorder in nanocrystalline materials manifests in ways reminiscent of fully amorphous materials, such as mesoscale shear localization and pressure-dependent yielding, owing to the high concentration of grain boundaries and their predominance in governing plasticity. Relaxation processes in nanocrystalline materials that facilitate reconfigurations of grain boundaries and lower their energy, such as low temperature annealing, have been shown to enhance mechanical strength. However, processes that raise the energy of a nanocrystalline metal have not been observed, limiting the tunability of properties and the prospect for suppressing shear localization. Here, we use femtosecond laser processing as a unique non-equilibrium process that can generate complex stress states due to...
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Journal: Science Advances
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Volume: Issue: First Page #:
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Article Title: A high-entropy alloy with hierarchical nanoprecipitates and ultrahigh strength
Authors: Zhiqiang Fu, Lin Jiang, Jenna L. Wardini, Benjamin E. MacDonald, Haiming Wen, Wei Xiong, Dalong Zh
Keywords: high entropy alloy; mechanical properties
Abstract: High-entropy alloys (HEAs) are a class of metallic materials that have revolutionized alloy design. They are known for their high compressive strengths, often greater than 1GPa; however, the tensile strengths of most reported HEAs are limited. Here we report a strategy for the design and fabrication of HEAs that can achieve ultrahigh tensile strengths. The proposed strategy involves the introduction of a high density of hierarchical intragranular nanoprecipitates. To establish the validity of this strategy, we designed and fabricated a bulk $Fe_{25}Co_{25}Ni_{25}Al_{10}Ti_{15}$ HEA to consist of a principal face-centered cubic (fcc) phase containing hierarchical intragranular nanoprecipitates. Our results show that precipitation strengthening, as one of the main strengthening mechanisms, contributes to a tensile yield strength (?0.2) of ~1.86 GPa and an ultimate tensile strength (UTS) of ~2.52 GPa at room temperature, which heretofore represents the highest strength reported for an HEA with an...
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Article Title: Combined effects of nonmetallic impurities and planned metallic dopants on grain boundary energy and strength
Authors: Zhifeng Huang, Fei Chen, Qiang Shen, Lianmeng Zhang, Timothy J. Rupert
Keywords: Segregation; Grain boundary energy; Embrittlement; Impurities; First-principles calculations
Abstract: Most research on nanocrystalline alloys has been focused on planned doping of metals with other metallic elements, but nonmetallic impurities are also prevalent in the real world. In this work, we report on the combined effects of metallic dopants and nonmetallic impurities on grain boundary energy and strength using first-principles calculations, with a ?5 (310) grain boundary in Cu chosen as a model system. We find a clear correlation between the grain boundary energy and the change in excess free volume of doped grain boundaries. A combination of a larger substitutional dopant and an interstitial impurity can fill the excess free volume more efficiently and further reduce the grain boundary energy. We also find that the strengthening effects of dopants and impurities are dominated by the electronic interactions between the host Cu atoms and the two types of dopant elements. For example, the significant competing effects of metal dopants such as Zr, Nb, and Mo with impurities on
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Article Title: Amorphous Intergranular Films Enable the Creation of Bulk Nanocrystalline Cu–Zr with Full Density
Authors: Olivia K. Donaldson, Timothy J. Rupert
Keywords: sintering, mechanically alloyed powder, nanocrystalline, Cu-Zr alloy, bulk nanostructured material
Abstract: Nanocrystalline metal alloys show great potential as structural materials, but are often only available in small volumes such as thin films or powders. However, recent research has suggested that dopant segregation and grain boundary structural transitions between states known as complexions can dramatically alter grain size stability and potentially enable activated sintering. In this study, we explore strategic consolidation routes for mechanically alloyed Cu-4 at.% Zr powders to capture the effects of amorphous complexion formation on the densification of bulk nanostructured metals. We observed an increase in density of the consolidated samples which coincides with the formation of amorphous intergranular films. At the same time, the grain size is reasonably stable after exposure to these temperatures. As a result, we are able to produce a bulk nano-grained metal with a grain size of 57 nm and a density of 99.8%, which shows an impressive balance of small grain size and high...
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Conference Location: Greece
Paper Title: Rejuvenation of Disorder-Containing Materials
Authors: GH Balbus, MP Echlin, CM Grigorian, TJ Rupert, TM Pollock, DS Gianola
Acknowledged Federal Support: **N**

PATENTS:

Intellectual Property Type: Patent Date Received: **29-Aug-2018**
Patent Title: Enhancing Mechanical Properties of Nanostructured Materials with Interfacial Films
Patent Abstract: Nanostructured materials that contain amorphous intergranular films (AIFs) are described hereir
Patent Number: 15/896,849
Patent Country: USA
Application Date: 14-Feb-2018 Application Status: 1
Date Issued:

Invited Lectures:

[L32] "Moving closer to equilibrium but maintaining the defects (and the properties)," *The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition*, February 2020, San Diego, CA.
[L31] "Making strong, tough, thermally-stable, and radiation tolerant nanocrystalline materials in bulk form," *The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition*, February 2020, San Diego, CA.
[L30] "Optimizing the mechanical behavior of metals with grain boundary and dislocation complexions," *International Conference on Plasticity, Damage, and Fracture*, January 2020, Rivera Maya, Mexico.
[L29] "The thermodynamics and kinetics of defect-driven complexion formation," *International Conference on Plasticity, Damage, and Fracture*, January 2020, Rivera Maya, Mexico.
[L28] "Using TEM to isolate the importance of complexion transitions on the behavior of nanocrystalline materials," *Second International Symposium on Advanced Microscopy and Spectroscopy (ISAMS-2)*, December 2019, Irvine, CA.
[L27] "Coupled experimental and computational studies of amorphous grain boundary complexions," *Materials Science & Technology (MS&T) Conference and Exhibition*, September 2019, Portland, OR.
[L26] "Linear complexion formation driven by local stress concentrations near dislocations," *Dislocations 2019*, September 2019, Haifa, Israel.
[L25] "Probing nanoscale complexion transformations with computational techniques that complement experiments," *Recent Advances in the Modeling and Simulation of the Mechanics of Nanoscale Materials Workshop*, August 2019, Philadelphia, PA.
[L24] "Segregation-Induced Complexion Transitions: New Opportunities for Materials Design," *Gordon Research Conference – Physical Metallurgy*, July 2019, Manchester, NH.
[L23] "Tailoring mechanical behavior with one- and two-dimensional complexions," *The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition*, March 2019, San Antonio, TX.
[L22] "Promoting beneficial complexion transitions: Tuning defect structure to make better materials," *University of California, Los Angeles – Department of Materials Science and Engineering*, March 2019, Los Angeles, CA.
[L21] "Promoting beneficial complexion transitions: Tuning defect structure to make better materials," *University of Illinois at Urbana-Champaign – Department of Materials Science and Engineering*, February 2019, Urbana, IL.
[L20] "Promoting beneficial complexion transitions: Using defects to make better materials," *Dartmouth College – Thayer School of Engineering*, January 2019, Hanover, NH.
[L19] "Enabling tough and stable nanocrystalline metals through the incorporation of amorphous complexions," *International Conference on Plasticity, Damage, and Fracture*, January 2019, Panama City, Panama.
[L18] "Decorating defects with segregating dopants to tailor mechanical properties," *Materials Science & Technology (MS&T) Conference and Exhibition*, October 2018, Columbus, OH.
[L17] "Dislocation-assisted linear complexion formation in body-centered cubic and face-centered cubic alloys," *Society of Engineering Science (SES) Annual Technical Meeting*, October 2018, Madrid, Spain. (KEYNOTE)

[L16] "Stabilization and toughening of nanocrystalline alloys through the incorporation of amorphous complexions," *THERMEC International Conference on Processing and Manufacturing of Advanced Materials*, July 2018, Paris, France.
[L15] "In situ mechanical testing of hierarchical and gradient nanostructures," *International Conference on Metallurgical Coatings and Thin Films (ICMCTF)*, April 2018, San Diego, CA.
[L14] "Promoting Beneficial Grain Boundary Phase Transitions with Segregation Engineering," *University of Pennsylvania – Department of Materials Science and Engineering*, April 2018, Philadelphia, PA.
[L13] "Manipulating the Structure and Properties of Nanocrystalline Metals using Segregation Engineering," *University of California, Berkeley – Department of Mechanical Engineering*, March 2018, Berkeley, CA.
[L12] "Competing effects of nonmetal impurities and planned metallic dopants on grain boundary deformation," *The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition*, March 2018, Phoenix, AZ.
[L11] "Stabilization of nanocrystalline alloys through the incorporation of grain boundary complexions," *The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition*, March 2018, Phoenix, AZ.
[L10] "Small-scale mechanical testing of hierarchical nanostructured materials," *The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition*, March 2018, Phoenix, AZ.
[L9] "Modeling of complexion transitions at one- and two-dimensional defects," *The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition*, March 2018, Phoenix, AZ.
[L8] "Decorating defects with segregating dopants to tailor mechanical properties," *Materials Science & Technology (MS&T) Conference and Exhibition*, October 2017, Pittsburgh, PA.
[L7] "Complexion transitions in metals: Unique opportunities for mechanical behavior and materials processing," *The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition*, February 2017, San Diego, CA.
[L6] "Controlling Nanocrystalline Structure and Properties with Segregation Engineering," *California Institute of Technology – Department of Mechanical and Civil Engineering*, February 2017, Pasadena, CA.
[L5] "Promoting Beneficial Grain Boundary Phase Transitions with Segregation Engineering," *University of California, Santa Barbara – Materials Department*, November 2016, Santa Barbara, CA.
[L4] "Using Grain Boundary Complexion Transitions to Toughen Nanocrystalline Metals," *University of California, Irvine - Department of Chemical Engineering and Materials Science*, October 2016, Irvine, CA.
[L3] "Adding Complexions to Nanostructured Metals to Achieve a Unique Combination of Strength and Ductility," *Functional and Nanomaterials 2025*, September 2016, Irvine, CA.
[L2] "Using Interfacial Structure to Control the Properties of Nanocrystalline Metals," *Sandia National Laboratories*, September 2016, Albuquerque, NM.
[L1] "Promoting Beneficial Grain Boundary Phase Transitions with Segregation Engineering," *University of Southern California – Materials Science and Engineering*, September 2016, Los Angeles, CA.

Contributed Talks *(Presenter's name is in bold)***:**

[T14] **Turlo V**, Rupert TJ. "Linear complexion formation and their effect on the strength of metallic alloys," *The Minerals, Metals and Materials Society (TMS) Annual Meeting & Exhibition*, February 2020, San Diego, CA.

[T13] Balbus GH, Echlin MP, Grigorian CM, Rupert TJ, Pollock TM, **Gianola DS**. "Controlling Disorder-Property Relationships in Metallic Alloys via Targeted Processing," *Society of Engineering Science (SES) Annual Technical Meeting*, October 2019, St. Louis, MO.

[T12] **Turlo V**, Rupert TJ. "Discovery of a wide variety of linear complexions in metallic alloys," *Materials Science & Technology (MS&T) Conference and Exhibition*, September 2019, Portland, OR.

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