Victoria Yi
‘28 McCormick School of Engineering
victoriayi2028@u.northwestern.edu
(667)-786-2735

Experimental Measurements of Grain Boundary Segregation in Binary Alloys

April 1st, 2025

ABSTRACT (250 words)

Grain boundary segregation describes the process by which atoms in a solute concentrate at the grain boundaries of a material, effectively changing the material’s properties. These nanocrystalline metals tend to have increased toughness, corrosion resistance, and ductility compared to their normal counterparts. While this phenomenon is known, few predictive models exist. Traditionally, TEM (transmission electron microscopy) and APT (atom probe tomography) have previously been used to create such models, however, these computational methods are severely limited by their extremely time-consuming nature and the resulting low volume of data points. This research aims to address the current lack of comprehensive experimental data on equilibrium grain boundary compositions for binary alloys by creating a complete data set for the Cu-Au system. This will be accomplished by creating homogeneous nanocrystalline Cu-Au samples through ball-milling, followed by annealing these samples at various temperatures and quenching to capture its equilibrium segregation states. The goal, a comprehensive experimental model, will be produced by directly measuring each sample’s physical constants through x-ray diffraction (XRD) and applying a conservation of solute approach, to calculate grain boundary composition. This comprehensive data set will make it possible to validate and refine existing computational models as well as increase understanding of the thermodynamic forces that drive grain boundary segregation. Additionally, further applications of this research could contribute to the development of advanced alloys with improved temperature resilience and resistance to failure, due to the increased accuracy of the predictive model for grain boundary segregation.

Where will this research occur? (50 words)

Primarily, this work will be done in the Schuh Group's lab space. However I'll also be fairly present in IMSERC and JB Cohen for x-ray diffraction purposes; IMSERC for transmission and JB Cohen for reflection.

Why will it produce new knowledge? (200 words)

This research will produce new knowledge by creating a complete data set of equilibrium grain boundary compositions for a model binary alloy. Currently, a data set of this size hasn't been achieved before outside of a few select embrittling systems. There are numerous computational models that focus on this topic on the atomistic scale, but without the experimental data to verify and calibrate these models significant ambiguity remains. A larger data set would make it possible for these existing models to be refined, uncovering more knowledge of the underlying thermodynamic processes that spur grain boundary segregation. With this knowledge, nanocrystalline metals could replace their bulk counterparts by engineering their grain boundaries for stability at extreme temperatures. Additionally, this research can lead to the reduction of cracking and failure in metal alloys with significant embrittlement at their grain boundaries, increasing the number of alloys capable of being used in advanced applications, like the aerospace industry.

RESEARCH SPECIFICS (750 words)

Within the space of grain boundary segregation research, there's a known lack of experimental data to back the results of atomistic computational models. TEM (transmission electron microscopy) and APT (atom probe tomography) have both been used to create individual experimental points to qualitatively confirm the trends found by the aforementioned models [1] . But these are specific points, not cohesive sets of experimental data. Additionally, these techniques are time consuming and rely on external calibrations, introducing more possibility for error. On the other hand, our group's method, detailed below, backs out the grain boundary composition directly through measuring physical constants. Thus, the goal of this research is to create a complete data set of equilibrium grain boundary compositions for a specific system (in this case, Cu-Au). Cu-Au is ideal for this application because it's a solid solution and relatively noble [2]. Consequently, there isn't much of a concern for oxide formation, which would negatively impact our ability to measure the grain boundary segregation of the sample. Additionally, the Cu-Au has previously been predicted to show significant segregation through the use of computational models [3]. Creating a large data set, utilizing the method below, will significantly increase our understanding of grain boundary segregation, a process that currently lies in a certain degree of ambiguity, due to a lack of knowledge of the tangible, thermodynamic values behind this process.

To create the Cu-Au samples, ball-milling will be utilized to ensure all the elements are combined into an atomically homogeneous compound, both inside and at the sample's grain boundary. This process of ball-milling will transform the sample into a nanocrystalline solid-state, making it possible to relax the sample's crystalline structure towards its equilibrium state with subsequent heat treatments. Ball-milling requires the precise weighing, mixing, and filling of elemental metal powders, along with the ceramic Al2O3, into Tungsten Carbide vials that will then be placed into the ball-milling machine. Various process parameters such as length of time, frequency (Hz), and number of cycles will be frequently adjusted to ensure that full homogenization (verified by X-ray diffraction) is reached for each different composition.

Once ball-milling is complete, the samples will be annealed at a given temperature to reach the equilibrium state of segregation, followed by quenching to prevent the sample from relaxing to a lower temperature. For this process, a furnace inside a glove box will be utilized to avoid exposure to oxygen. Each unique sample will be divided into three crucibles and placed into the furnace at a high temperature (like 600*C). Every one or two hours, the temperature will be lowered by 100*C and one crucible will be removed; this process will repeat until there are no crucibles remaining. Both the use of annealing and quenching will expose how temperature and time impact the amount of segregation in the sample.

To collect the diffraction pattern of these quenched samples, x-ray diffraction will be done in the JB Cohen laboratory space, using the Rigaku Ultima machine. The resulting XRD data will be analyzed through a process called Rietveld refinement, where a theoretical line profile is matched to the XRD data to simultaneously fit the entire pattern by least-squares refinement. Rietveld refinement is the most preferable form of refinement due to its efficiency and highly accurate quantitative analysis [4]. This will accurately reveal each sample's lattice parameter and grain size; utilizing these values, the sample's true composition will be calculated through a modified vegard's law. Determining the composition validates the sample's accuracy and highlights any abnormalities in the data. The aforementioned data will also be graphed to investigate trends between final quench temperature, lattice parameter, grain size, and grain boundary values.

These diffraction patterns are key to calculating the composition of the original sample; this is necessary to validate its true composition, and the sample's fitness for later procedures. From the calculated composition, the amount of solute that's moved into the sample's grain boundary can be calculated using a conservation of solute approach, and ultimately graphed against final quench temperature [C]. This method will be scaled and utilized to create hundreds of grain boundary values, and thus, a complete set of equilibrium grain boundary compositions for Cu-Au. Finally, this data will be fitted to numerous thermodynamic equations to derive the relevant variables.

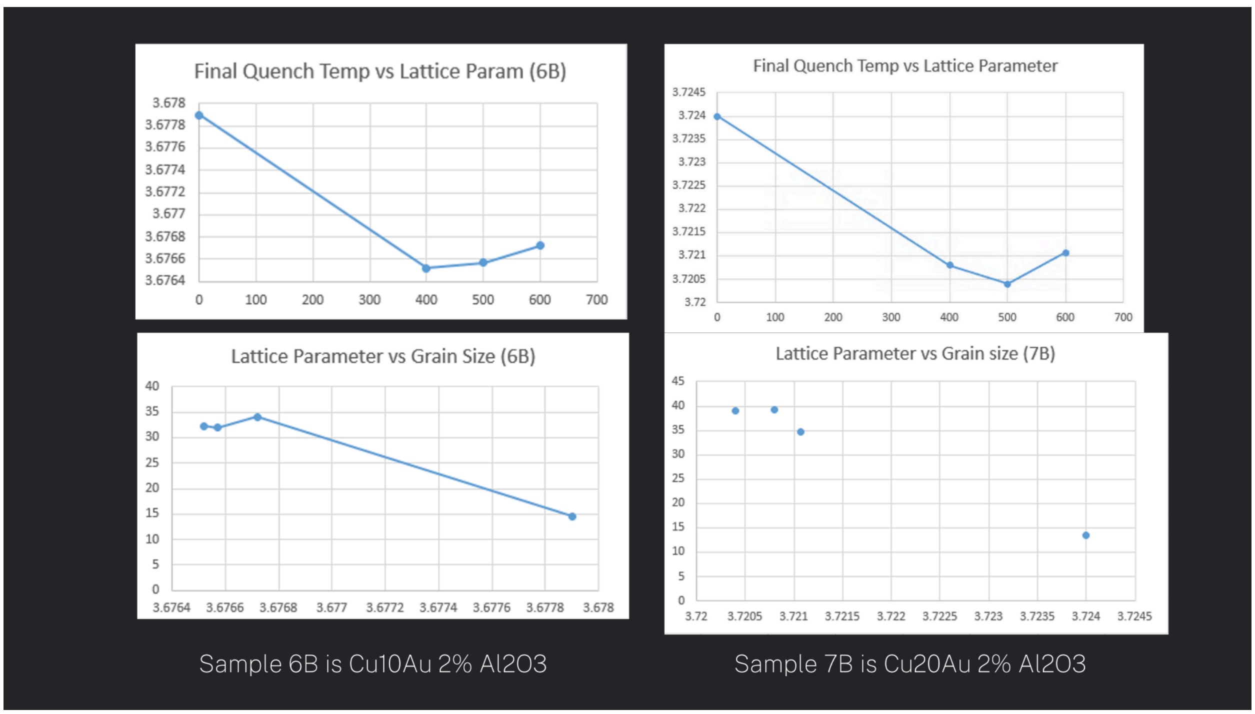
1. P. Lu, F. Abdeljawad, M. Rodriguez, M. Chandross, D.P. Adams, B.L. Boyce, B.G. Clark, N. Argibay, “On the thermal stability and grain boundary segregation in nanocrystalline PtAu alloys”, Materialia, Volume 6, 2019

2. Okamoto, H., Chakrabarti, D.J., Laughlin, D.E. *et al.* The Au–Cu (Gold-Copper) system. *JPE* 8, 454–474 (1987). https://doi.org/10.1007/BF02893155

3. Malik Wagih, “Spectrum of Grain Boundary Segregation Energies in a Polycrystal”, 2019

4. S.A. Hassanzadeh-Tabrizi, “Precise calculation of crystallite size of nanomaterials: A review”, Journal of Alloys and Compounds, Volume 968, 2023

Images of Cu-Au Data I’ve Collected:

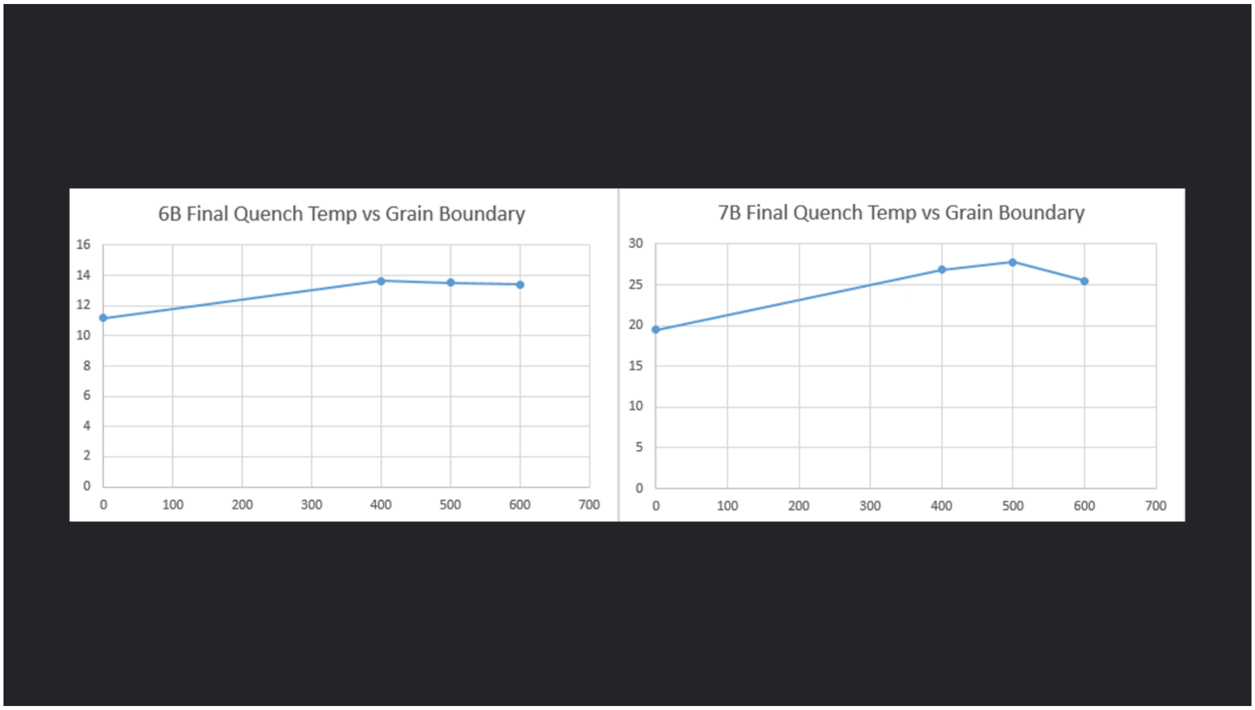


Sample 6B is Cu10Au 2% Al2O3

Sample 7B is Cu20Au 2% Al2O3

A. Sample 6B & 7B comparisons between final quench temperature (*C) and lattice parameter.

B. Sample 6B & 7B comparisons between lattice parameter and grain size (nm).



C. Sample 6B & 7B comparisons between final quench temperature (*C) and corresponding calculated grain boundary values.

THE VALUE OF THE REWARD (250 words)

As a student who comes from a rural, small town background, I'd never even touched a glove box before I came to Northwestern. I was never exposed to this idea that research could be more than just mixing chemicals together and seeing if something happens. In reality, at Northwestern, I learned that research manifests in many different ways and that anyone can do research— just as long as they have an idea. This reward would give me the ability to see through a project of my own and gain valuable insight on how to independently solve problems and create new knowledge. The reward would make it possible for me to stay in Evanston over the summer and participate in a research opportunity like nothing I've ever done before. And as a FGLI student, I know that these kinds of opportunities, paired with financial support, are few and far between. Even the chance alone, to expand on an idea, to turn it into knowledge that's new and valuable, is so incredibly valuable in and of itself.

RESUME

Victoria Yi

Manufacturing and Design Engineering

INTERNSHIPS

Science & Engineering Apprenticeship Program; Physics Dept.

US Naval Academy · Summer 2022

- 40 hrs/week for 8 weeks; Prof. Murray Korman & Kinear Chair Jennifer Bartlett
- Built a makeshift sextant (<$5); studied its precision by calculating my coordinates
- Created an environment sensor/logger for the US Coast Guard Academy's library
- Small projects: laser refraction study, captured images of celestial objects, created educational games about astronomy for Fairfax County Fair (VA)

ASPIRE Internship; Applied Physics (AOS/QLC)

Johns Hopkins University Applied Physics Lab · Summer 2023

- 200+ total hours; Dr. Christopher Lavelle
- Constructed a fully functional gamma ray spectrometer out of low-cost (<$300) hardware
- Programmed in C and Python for the spectrometer to do scintillation detection and instrument calibration

RESEARCH

Dean Schuh Group Undergrad Research Assistant

Northwestern University · McCormick School of Engineering · Present

- 10-15 hours per week; mentored by Jacob Mack & Prof Schuh, Dean of McCormick School of Engineering
- Researching nanocrystalline alloys and how to stabilize them via grain boundary segregation
- Experience with ball-milling, StadiP, Topas, and GSAS
- Frequent use of glove box and furnace

The Protractor, The Sextant, and The Student iPoster

American Astronomical Society's 241st Meeting · January 9, 2023

- Citation: American Astronomical Society Meeting #241, id. 170.06. Bulletin of the American Astronomical Society, Vol. 55, No. 2 e-id 2023n2i170p06
- Created an abstract and an iPoster; mentored by Dr. Jennifer Bartlett
- Focus: build a sextant out of common materials and test its accuracy by using celestial navigation principles to find my apparent latitude and longitude
- Learned: find local apparent noon, measure the sun's zenith distance, use the 2022 Nautical Almanac, calculate GHA, compare coordinates with percent error

EMPLOYMENT

Larriland Farm Employee

Larriland Farm · Summer 2024

- Helped customers select produce, answered questions, and shared knowledge on produce and ripeness
- Managed the barn's inventory (perishable & shelf-stable), processed shipments from local businesses, and frequently labelled & restocked goods
- Processed customer payments and assisted customers with parking
- Swept floors, disinfected food surfaces, and took out the trash daily

CottonStitchPlus Etsy Shop

Home Business · April 2020- Present

- Founder & owner; 1900+ total sales
- Donated 650+ masks to local institutions (like Johns Hopkins Hospital) during the pandemic
- Sold custom water bottles, fabric keychains, t-shirts, and cotton masks
- Featured in CityBox Maryland; package of products from small businesses in MD

CONTACT

667-786-2735
VictoriaYi2028@u.northwestern.edu
Woodstock, MD

EDUCATION

Northwestern University

Materials Engineering Major

Class of 2028 · Creative Writing Minor

Marriotts Ridge High School

2020-2024 · Unweighted GPA: 4.0

Weighted GPA: 4.89

SKILLS

Python · C · JavaScript · Excel · AutoCAD · ArduinoIDE · Soldering · RaspberryPi · Circuit-building

AWARDS

National Merit Scholarship Program

2023 National Merit Finalist

Scholastic Art & Writing Awards

2023 Gold Key Award; Writing

National Honor Society

Student Member (2022- Present)

Genius Olympiad

2022 Finalist; Science category

CollegeBoard National Rural and Small Town Recognition Program

2023 Awardee

CREATIVE PUBLICATION

Yi, V. (2024). HAMARTIA. In Heiliger, E. & DuPuis, D. (Eds), Navigating the Margins: A Collection of Stories by High School Students (pp. 284-302). BookBaby."

COMMUNITY SERVICE

Scholastic Book Program at Veterans Elementary School

October 2021- 2023

Helped teachers earn free books through the Scholastic Book Club program by creating packets of Scholastic flyers & cover letters for them every season.