Resolving Pseudosymmetry in γ-TiAl using Cross-Correlation Electron Backscatter Diffraction with Dynamically Simulated Reference Patterns

HONORS THESIS PROPOSAL

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Statement of Intent

Investigate the effectiveness of using high-resolution electron backscatter diffraction with dynamically simulated reference patterns in resolving pseudosymmetry.

Introduction

Electron backscatter diffraction (EBSD) is a common method of analyzing the microstructure of materials composed of crystalline lattices. By bombarding a material sample with an electron beam, diffracted electrons that exit the sample surface are captured on a phosphor screen. The resulting electron backscatter diffraction pattern (EBSP) can be analyzed to obtain information about the crystal structure and orientation. By scanning areas of the sample and collecting EBSPs at multiple locations, the orientation information obtained from EBSP analysis can be used to identify grains in polycrystalline materials, analyze grain boundary characteristics, and perform texture analysis. Traditional EBSP analysis techniques utilize Hough transforms to identify band positions. The Hough transform maps a line in an image to an intercept and angle on a scatter plot; EBSD bands are transformed to "Hough peaks" in Hough space that are indexed with a simulated set of

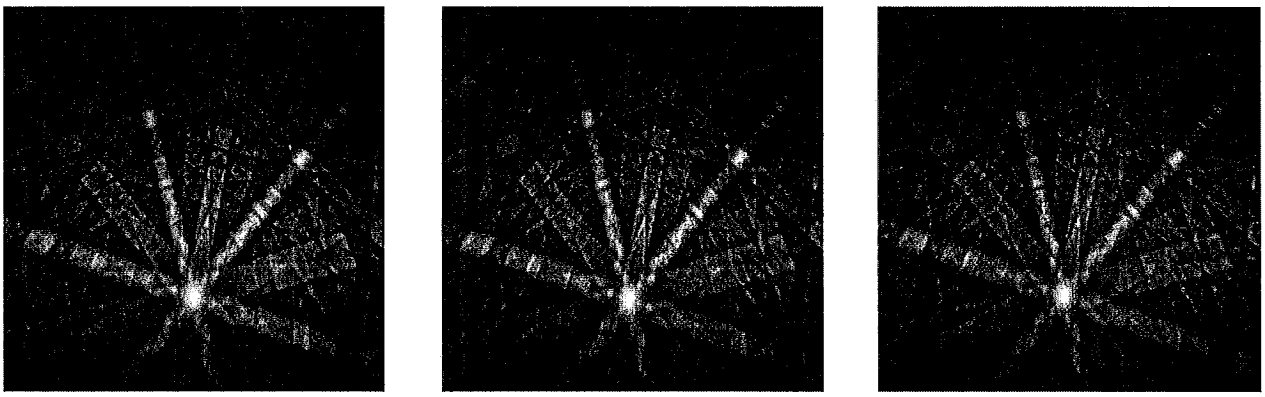

Figure 1 Dynamically simulated EBSD patterns of sample pseudosymmetric orientations of γ-TiAl

Hough peaks based upon the specified lattice structure. The term "indexing" is often used to refer to the process of using Hough transforms to determine the microstructural characteristics of an EBSP. Automated methods for indexing EBSPs are commonly used, often in software directly tied to the scanning electron microscope (SEM) used to collect the patterns.

Currently, automated indexing algorithms have difficulty resolving pseudosymmetry. Pseudosymmetry occurs when two patterns have nearly identical band positioning, but unique crystallographic orientations, as shown in Figure 1, where to the eye the patterns appear identical, but have subtle differences that need to be resolved. Pseudosymmetry is most commonly found in materials where one side is slightly longer than the other two of equal length. Tetragonality is the measurement of relative elongation of the longer side. Titanium aluminides, commonly used in commercial aero-engines, and martensite, an extremely hard phase of steel, are common engineering materials with a tetragonal structure that exhibit pseudosymmetry. Resolution of pseudosymmetric orientations in these materials is important in identifying the slip systems that uniquely contribute to strength and hardening characteristics of the material.

Several approaches have been used to resolve pseudosymmetry. Zambaldi et al proposed using a fit characteristic of the indexing algorithm to correctly identify orientations in pseudosymmetric materials. This approach achieved a successful indexing rate up to 90%. Other recent approaches have used HR-EBSD techniques to attempt to resolve pseudosymmetry.

High-resolution EBSD (HR-EBSD), or cross correlation EBSD, improves upon traditional Hough methods by comparing sub-regions of the EBSP with a reference EBSP. The shifts required to align the two regions are calculated using Fast Fourier Transforms (FFT), which are then used to calculate the relative distortion and orientation between the two patterns. This technique offers excellent relative orientation determination, several orders of magnitude better than Hough techniques.

Within the HR-EBSD technique exist two fundamental approaches for selecting the reference pattern. The traditional method selects a pattern from the same scan as the pattern of interest, typically from a location of low or near-zero strain, from a region far from any edges or defects, or from a centralized location in the grain. The second and more recently developed approach consists of generating a simulated reference pattern. Since the distortion and orientation of the simulated pattern are precisely known, the relative distortion and orientation information generated by the cross-correlation can theoretically be used to determine the absolute distortion and orientation of the experimental pattern. The determination of absolute distortion allows for calculation of absolute strain and tetragonality, which can potentially be used to resolve pseudosymmetric orientations based upon the identification of the elongated tetragonal axis, or *c*-axis.

Additionally, several methods exist within HR-EBSD to calculate a quantitative measure of fit, or correlation, between the experimental pattern and the reference pattern. Although pseudosymmetric orientations have nearly identical band positions, their band intensity profiles —composed of band intensities and band widths —are unique, such that they can often be resolved by simple visual inspection. Therefore the pixel-by-pixel comparisons used to calculate these measures of correlation between the experimental and reference images may be another potential method of resolving pseudosymmetry.

Since the absolute orientation of the reference pattern must be known in order to identify the correct pseudosymmetric orientation, this restricts the method to using simulated reference patterns. There are

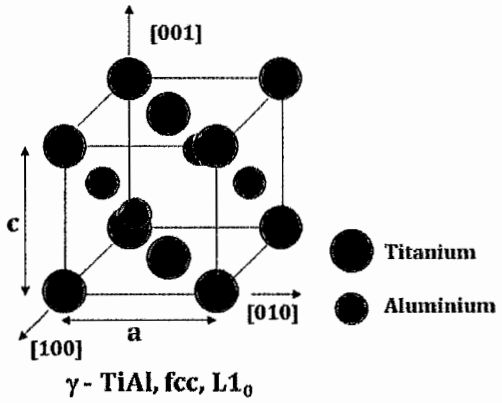

Figure 2 Crystal structure of γ -TiAl phase. Courtesy of [7]

currently two principal methodologies used to generate simulated EBSPs: kinematical simulation and dynamical simulation. Kinematical simulations are simplistic simulations based upon Braggs Law, which defines positions for which constructive interference of scattered electrons occurs from planes of a specified interplanar spacing. Kinematical simulations accurately replicate band positions, but they suffer from poor band profile and intensity replication, especially near band axes. However, due to the simplistic nature of the simulation, they can be generated very quickly using efficient algorithms, typically in under a second. Dynamical simulations, on the other hand, are based upon a Monte Carlo simulation which computes the trajectories of individual electrons as they interact with the crystal and undergo scattering events. By simulating the trajectories of several million electrons, a high-fidelity EBSP can be generated that accurately replicates band position, profile, and intensity. The Monte Carlo simulation is extremely computationally intensive, often requiring about 30 minutes to complete; however, once the initial "master" EBSD pattern is generated by the Monte Carlo simulation, individual orientations of the material can be quickly generated. Since accurate simulation of band profiles are likely very important in resolving pseudosymmetry, dynamically simulated reference patterns are the better option.

Accurate determination of microscope geometry presents a significant challenge when using simulated reference patterns. The pattern center of an EBSP, defined as the location where the electrons impinge normal

to the surface of the detector screen, can have a significant effect on calculation of absolute strain and tetragonality. If the pattern center is incorrect, the reference pattern will be slightly shifted relative to the experimental pattern, resulting in artificial strain between the two patterns. Since pseudosymmetric orientations so closely resemble each other, it is imperative that the pattern center error is small, otherwise an incorrect orientation may be selected as the correct one. It is important that the sensitivity of the above methodologies to pattern center error also be established and that methods be developed to minimize or eliminate the influence of pattern center error on resolution of pseudosymmetry.

The purpose of the current paper is to investigate the effectiveness of using dynamically simulated reference patterns to resolve pseudosymmetry in a common engineering material. This will include using HR-EBSD techniques to calculate tetragonality of the experimental pattern, as well as using measures of correlation between the patterns. Additionally, the effects of pattern center on the accuracy of this resolution will be evaluated.

Methodology

To determine the effectiveness of using dynamically simulated reference patterns to resolve pseudosymmetry, both a simulated scan and an experimental scan of γ-TiAl will be analyzed. Due to its low density, nearly constant yield strength up to 1073 K, and good corrosion and creep resistance, γ-TiAl is an excellent choice of material in high-temperature structural components, such as in jet engines and turbines. The structure of the γ-TiAl phase is a face-centered cubic cubic-derived tetragonal lattice. The *c*-axis is about 2% longer than the *a*-axis due to alternating layers of Ti and Al occupying the (002) planes. Resolution of pseudosymmetry due to the slight tetragonality will aide in better characterizing deformation modes and therefore strength and hardening characteristics in this important engineering material.

The initial analysis will be performed using a dynamically simulated scan of TiAl. The simulated patterns will be generated by EMsoft 3.0, an open-source software package for simulation of electron diffraction and

imaging modalities developed at Carnegie Mellon University. Since the orientation, lattice parameters, and microscope geometry can be exactly specified using simulated patterns, a controlled experiment investigating the ability of dynamically simulated reference patterns to resolve pseudosymmetry can be performed.

The simulated scan will consist of 10 grains composed of 10 patterns each. The orientations within each scan will vary in orientation by 1 degree over the 10 points. Since pseudosymmetric orientations for γ-TiAl come in trios, three sets of pseudosymmetric orientations will be included, with one random orientation. The orientations will be arranged such that one set of grains are all adjacent, the other set will have two adjacent grains, and the third will have all three grain separated (see Figure 3). The pattern center will be varied with a common step size for all scan points. The sample tilt and azimuthal, camera tilt and azimuthal, accelerating voltage, and phosphor screen size will be kept at their default values.

Once the simulated scan is generated, the scan will be indexed using OIM Data Collection, software developed by EDAX. It is expected that the indexing will not be able to resolve all of the orientations. The results of the indexing will then be analyzed using OpenXY, a proprietary open-source software developed by Brigham Young University to perform HR-EBSD analysis using real, kinematically simulated, or dynamically simulated reference images.

The first approach will be to use OpenXY to calculate the absolute strain of the images using dynamically simulated reference images. The tetragonality of each point can then be determined and used to identify the orientation of the *c*-axis, which will identify the correct pseudosymmetric orientation. The second approach will be to modify OpenXY use EMsoft to generate a dynamically simulated reference pattern for the current orientation, a 90 degree rotation about the *a*-axis, and a 90 degree rotation about the *b*-axis. The correlation of each reference pattern with the pattern of interest will then be calculated using various parameters, including

Figure 3 Layout of simulated scan. Each color represents a unique set of pseudosymmetric orientations.

the cross-correlation coefficient, mutual information, shift confidence, and norm of the deformation gradient tensor. The reference image with the highest degree of correlation using any one or all of these parameters will then be selected as the correct orientation.

The effectiveness of the methodology will be verified by comparing the corrected orientations with the original orientations used to generate the scan.

Once this initial analysis has been completed, the same methods will be applied to an experimental sample of γ-TiAl. The sample to be used is an existing sample of lamellar γ-TiAl used in previous experiments by Brigham Young University. The sample has been prepared for inspection by cleaning it with an ultrasonic cleaner, followed by polishing the surface with diluted colloidal silica. A scan of the sample will be taken using an SEM microscope at Brigham Young University, and the patterns will be indexed and saved for post-processing.

The same process described above will be repeated for the experimental scan to determine how well the methods can be applied to experimental methods. The effectiveness of method will be evaluated by measuring the number of scan points within a grain that are dissimilar to those surrounding them. Alternatively, a measure of the entropy of the orientation within the grain may be used.

In order to analyze the effect of pattern center error on resolution of pseudosymmetry using dynamically simulated reference patterns, a new simulated scan will be generated using only one image from each of the ten grains in the original simulated scan. These ten images will be analyzed after varying the pattern center independently along the x, y, and z axes. The effect of increasing pattern center error will be determined by analyzing the number of failed orientation corrections. It is expected that pattern center error will be tolerable below a certain threshold.

Subsequently, the existing pattern center calibration methods will be validated by calibrating patterns with increasing values of pattern center error, in order to determine the maximum value of initial pattern center error that can be corrected by the calibration technique.

Proposed Outline

1. Introduction
2. Methodology
    2.1. Simulated Scan Generation
    2.2. Experimental Scan Preparation
    2.3. Sample Analysis
        2.3.1. Pattern Center Calibration
        2.3.2. Determination of tetragonality
        2.3.3. Pattern-matching using measures of correlation
3. Results and Discussion
4. Conclusion

Preliminary Research

[1] C. Zambaldi, S. Zaefferer and S. I. Wright, "Characterization of order domains in gamma-TiAl by orientation microscopy baed on electron backscatter diffraction," *Applied Crystallography,* no. 42, pp. 1092-1101, 2009.

Approach to resolving pseudosymmetry in γ-TiAl by detection of the minor tetragonal distortion of the diffraction patterns by an accurate measurement of band positions. Describes methods for accurately

determining pattern center. Uses a Hough space-based fit measure to determine correct orientation. Achieved 90% success rate. Validated with superlattice reflections.

[2] G. Nolze, A. Winkelmann and A. P. Boyle, "Pattern matching approach to pseudosymmetry problems in electron backscatter diffraction," *Ultramicroscopy,* no. 160, pp. 146-154, 2016.

Use of simulated patterns to resolve framboidal pyrite. Used cross-correlation coefficient to distinguish correct orientations.

[3] S. Dey, A. Morawiec, E. Bouzy, A. Hazotte and J. Fundenberger, "A technique for determination of γ/γ interface relationships in a (α2 + γ) TiAl base alloy using TEM Kikuchi patterns," *Materials Letters,* no. 60, pp. 646-650, 2006.

Analysis of lamellar gamma TiAl with TEM using superlattice band detection.

[4] B. Simkin, B. Ng, T. Bieler, M. Crimp and D. Mason, "Orientation determination and defect analysis in the near-cubic intermetallic γ-TiAl using SACP, ECCI, and EBSD," *Intermetallics,* no. 11, pp. 215-233, 2003.

Corrections to EBSD orientation maps for gamma TiAl by detecting superlattice reflections using selected area channeling patterns (SACP) in conjunction with electron channeling contrast imaging (ECCI).

[5] S. Dey, E. Bouzy and A. Hazotte, "EBSD characterisation of massive γ nucleation and growth in a TiAl-based alloy," *Intermetallics,* vol. 14, no. 4, pp. 444-449, 2006.

Analysis of nucleation and growth in gamma TiAl. States that EBSD is unable to differentiate the six possible gamma variants. FCC used instead.

[6] Oxford Instruments, "EBSD Analysis of TiAl alloys for texture and interphase boundary analysisq," 2010.

Application note stating that Oxford Instruments Channel 5 software can resolve pseudosymmetry in TiAl using an "Advanced Fit" method. Uses a refinement of the Hough transform. Sounds similar in scope to [1].

Qualifications

I have been researching electron backscatter diffraction techniques for almost two years under the supervision of Professor David Fullwood in the Mechanical Engineering Department at Brigham Young University. I am the primary author of "Performance of dynamically simulated reference patterns for cross correlation EBSD," which will be submitted shortly for peer review and publication. This paper discusses the differences between kinematically and dynamically simulated reference images when performing HR-EBSD analysis on a standard reference material. I am also co-author of two unpublished papers, "Characterization of Dislocation Motion Across Grain Boundaries in Commercial Pure Tantalum" and "Influence of Noise Generating Factors on Cross Correlation EBSD Measurement of GNDs." My role of both of these papers is tied to my role as primary developer of OpenXY, a proprietary open-source software for performing HR-EBSD analysis. I have been developing OpenXY over the past two years and have been primarily responsible for releasing it open-source, improving user interface, fixing runtime errors, expanding compatibility with various scan formats, and integration with EMsoft 3.0 developed by Carnegie Mellon University.

I am extremely familiar with HR-EBSD and simulated reference patterns, and am well qualified to investigate the use of dynamically simulated reference images to resolve pseudosymmetry.

Qualifications of Advisor

Dr. David Fullwood has been supervising my research in HR-EBSD for the past two years, and been publishing papers in microstructure-sensitive design, electron microscopy, and nanocomposites at BYU since 2005.

Schedule

- June 13, 2016: Finish analysis of simulated scan
- July 1, 2016: Scan of sample material completed
- July 15, 2106: Analysis of experimental sample completed
- August 1, 2016: Start rough draft
- August 29, 2016: Finish rough draft
- October 3, 2016: Second draft (after review by faculty advisor and two other peers in the same area of expertise)
- October 24, 2016: Polished draft (after two additional reviews)
- December 12, 2016: Final draft submitted
- February 12, 2017: Thesis submitted to Honors Program
- March 11, 2017: Thesis defense completed
- April 20, 2017: Final thesis copy uploaded

Resources

[1] C. Zambaldi, S. Zaefferer and S. I. Wright, "Characterization of order domains in gamma-TiAl by orientation microscopy baed on electron backscatter diffraction," *Applied Crystallography,* no. 42, pp. 1092-1101, 2009.

[2] G. Nolze, A. Winkelmann and A. P. Boyle, "Pattern matching approach to pseudosymmetry problems in electron backscatter diffraction," *Ultramicroscopy,* no. 160, pp. 146-154, 2016.

[3] S. Dey, A. Morawiec, E. Bouzy, A. Hazotte and J. Fundenberger, "A technique for determination of γ/γ interface relationships in a (α2 + γ) TiAl base alloy using TEM Kikuchi patterns," *Materials Letters,* no. 60, pp. 646-650, 2006.

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[5] S. Dey, E. Bouzy and A. Hazotte, "EBSD characterisation of massive γ nucleation and growth in a TiAl-based alloy," *Intermetallics,* vol. 14, no. 4, pp. 444-449, 2006.

[6] Oxford Instruments, "EBSD Analysis of TiAl alloys for texture and interphase boundary analysisq," 2010.

[7] F. Iqbal, "Fracture Mechanisms of γ–TiAl Alloys Investigated by In-situ Experiments in a Scanning Electron and Atomic Force Microscope," 2012.