TITLE: Application of a Crystal Orientation Method to Forensic Physical Matching of Metal Surfaces Along a Fracture Line

COLLABORATING INVESTIGATORS:

Dr. Barbara K. Lograsso
Michigan Technological Univ.
Mechanical Eng. Technology
Houghton, MI 49931
Phone:(906) 487-3658
Email: bklogras@mtu.edu

Dr. Thomas A. Lograsso
Ames Laboratory
126Metals Development
Ames, IA 50011
Phone: (515) 294-8425
Email: lograsso@ameslab.gov

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Table of Contents Page

INTRODUCTION 3
Forensic Need 3
Approach to address the problem 3
PROJECT OBJECTIVES: 3
TECHNIQUE 3
Crystal Orientation 3
EBSD background. 4
MATERIALS AND METHODS 6
304 Stainless Steel 7
Iron-gallium alloy 7
SEM and EBSD analysis 7
RESULTS AND DISCUSSION 9
304 Stainless Samples 9
Iron – Gallium Alloy Samples 12
Uniqueness of linear sequences 14
SUMMARY AND CONCLUSIONS 15
DISSEMINATION 15
APPENDIX 17

INTRODUCTION

Forensic Need

There is a need for a method that provides a quantitative measure of the nature and characteristics of materials with a known error rate that can be applied to performing physical matches. This study examined the possibility of using surface crystal orientation to associate, or differentiate, metal fracture fragments. This study investigated the feasibility of the Electron Backscatter Diffraction (EBSD) techniques to determine crystallography of the fracture and, in combination with conventional forensic methods, to physically associate broken surfaces of metal pieces to assess whether the broken pieces were at one time one piece. This study seeks to provide an additional method to establish individual characteristics of a fracture based on atomic arrangements, and it is possible that the high degree of resolution that this instrument will bring to bear on this problem may contribute new information to the ongoing debate about the uniqueness of fracture patterns. This work was conducted at the Ames Laboratory in collaboration with Jim Kreiser, retired tool mark examiner from the Illinois State Police.

Approach to address the problem

Conventional matching of the fracture surfaces utilizes physical characteristics of the fracture such as shape, color, and other features, and while quite useful, these features unfortunately do not lend themselves to an objective or quantitative evaluation of the uniqueness of a fracture. Our approach is to probe the atomic arrangement of atoms of individual and combination of metal crystals within and along a metal. The atomic arrangements of atoms are precisely known and can be determined to a high degree of accuracy. This study sought to test if crystal orientations of the fractured crystals across the fracture plane for two matching surfaces (as determined by a forensic examiner to be a matching fractures by conventional methods) are in fact, unique and a reliable, quantitative measure to add objectivity to the matching process.

PROJECT OBJECTIVES:

The overall objective of this study was to evaluate whether surface crystal orientation can be used to associate metal fracture fragments. This study sought to test if the orientations of the fractured crystals across the fracture plane for two surfaces determined by a forensic examiner to be a matching fracture by conventional methods. This study used Electron Back-Scattered Diffraction (EBSD), sometimes known as Orientation Imaging Microscopy (OIM), to determine the crystallographic orientation of individual metal crystals along the length of the fracture on a surface perpendicular to the actual fracture surface. This investigation examined the uniqueness of crystal orientations within a metal and examined the requirements necessary for accurate determination of crystallography using EBSD. This study also examined the crystallographic information as to whether it is sufficiently reliable characteristic from which a quantitative determination could be made that two separate pieces of metal are, in fact, from a single piece.

TECHNIQUE

Crystal Orientation

Metals are composed of crystals whose atoms are arranged in regular long range periodicity. This long range order is known as a crystalline state. The metals used in this study have a cubic crystal structure meaning that the atoms are position at lattice positions within a unit cubic cell. This unit cubic cell is repeated throughout the crystal and is the smallest unit which can completely describe

each atom's position. Most metals are composed of many crystals and in this context; crystals are referred to as grains and the collection of crystals as the grain structure. Grains within a metal have identical unit cells and chemical composition, but differ in the rotation or orientation of the unit cell relative to each other. This mismatch in orientation between two crystal lattices defines where two grains meet, and is called a grain boundary. It is this relative misorientation between adjacent grains that is the relevant to this study.

The relationship between grain orientations is commonly expressed as axis-angle pairs [1]. Imagine cube A is positioned next to cube B and cube B is randomly rotated with reference to cube A. There is an axis which is common to a crystallographic plane for both cubes. A rotation of cube B about this axis will bring it into coincidence with cube A. There are twenty-four different rotation angles possible for cube B due to cubic symmetry. The smallest angle which brings cube B into coincidence with cube A is known as the misorientation angle [2]. The misorientation angle between randomly oriented cubic crystals follows a Mackenzie distribution [2, 3]. The Mackenzie distribution has a mean rotation angle of 40.74° and a maximum rotation angle of 62.80° [3]. This study will use this misorientation angle in describing the relative changes in the crystallography of each grain along the fracture surface.

EBSD background.

Orientation measurements are made using electron back scattered diffraction (EBSD). EBSD is a point to point (crystal to crystal) measurement on the surface of the metal. The basic operation of EBSD is to collect electron back scattered patterns (EBSP) using a specialized detector on a Scanning Electron Microscope (SEM) as represented schematically in Figures 1 and 2. Orientation patterns are generated by how electrons interact with planes of atoms within the metal crystal (Figure 1). Kikuchi bands are generated by sample interaction with the electron beam (Figure 1). The band intensity is calculated using a kinematical electron diffraction model which is a function of the structure factor for crystal diffraction plane (hkl) and a function of the atomic scattering factor of each element, lattice plane indices, and atom positions. The band width (twice the Bragg diffraction angle) is also a function of the accelerating voltage (V) and the crystal lattice parameter (a). Examples of EBSP for differing orientation are given in Figures 1 and 2. Collected EBSP patterns are analyzed by comparison to simulated patterns based on the crystal known cubic structure of the metal. Angular differences in band location, band intensity, and band width differences between patterns allow for the unique identification of crystal orientation. The clarity of the patterns and hence, the ability to determine the orientation is affected both by the degree of deformation and surface quality since the patterns are generated from the very surface of the metal.

EBSP patterns can be collected automatically at regularly spacings across a pre-designated area that can encompass both side of a fracture. At each location, a specific orientation of the crystal can be determined from the pattern collected and a confidence index number to each point is assigned based on its confidence that the diffraction pattern was indexed correctly. The value of the confidence index is between zero and one with one being the highest confidence. This confidence index is dependent on the deformation and quality of polish, i.e. the overall surface quality at that location. Although a single data point is sufficient to describe the crystallography of any given grain, it is typical to have multiple points within a grain for statistical reasons. The distance the beam moves between each point is known as the step size and set by the operator to be less than size of the grain. The beam then rasters over the chosen area and orientation information is collected for all points within the area. This complete data set is then used for orientation maps and other data analyses such as line scans used in this study.

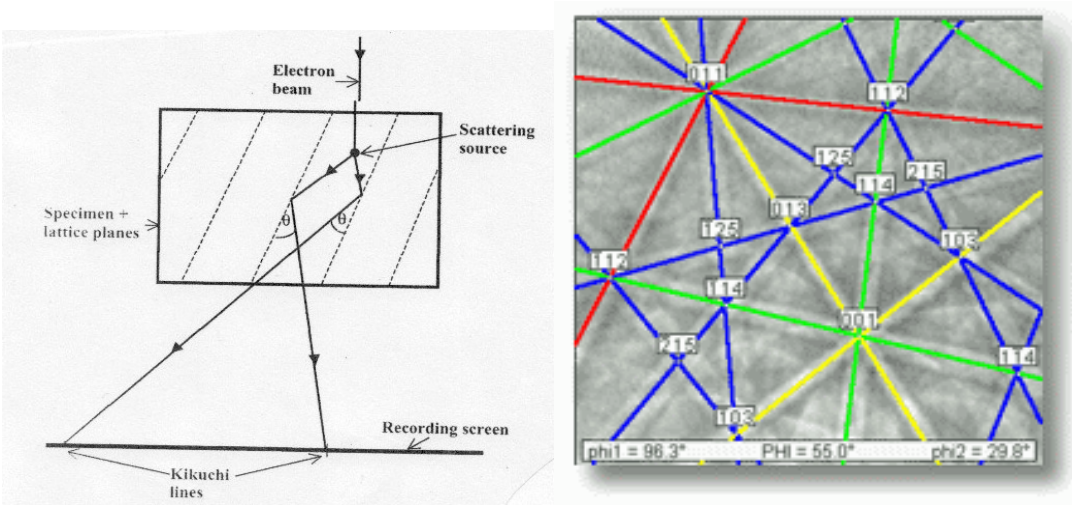
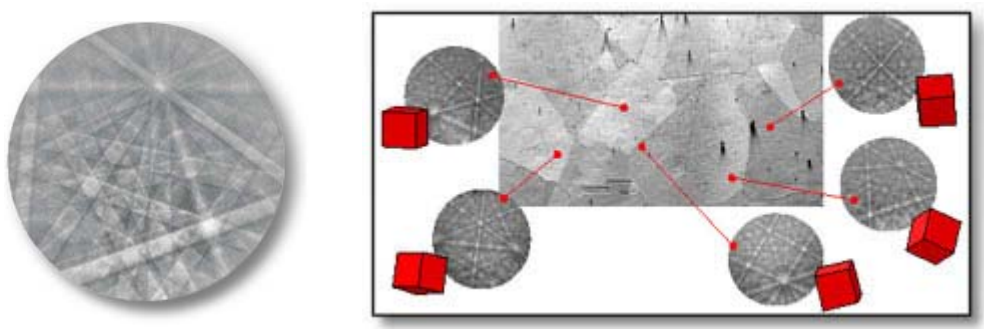


Figure 1. Electrons are channeled by lattice planes within the crystal (left) and create Kikuchi lines on a recording screen. These lines appear as bands on the recording screens. The intersections of Kikuchi bands (right) are indexed by calculating the angular distances between intersections and comparing them to known angles for a cubic unit cell. The indexing is automated allowing for the fast collection of crystal orientations.



Kikuchi Bands

Figure 2. Backscattered electron patterns are collected from positions on sample surface [1] and an example of various orientations of adjoining crystals on the metal.

There are two main ways to visualize the orientations of a collection of grains within a given piece of metal. For a collections of grains within a metal, an Orientation Distribution Function (ODF) can be generated which describes crystallite orientations fully within a specimen. ODFs are a common technique used in texture analysis to represent distributions of crystal orientations in relation to the macroscopic sample orientation (e.g. rolled sheet). ODF analysis involves basically rotating one of the coordinate systems about various axes until it comes into coincidence with the other. Rotations are applied in the following order for 3 Euler angles (φ_1, Φ, φ_2): 1) rotation of φ_1 about Z-axis, 2) rotation of Φ, about the X-axis, and 3) rotation of φ_2 about the Z-axis as shown in Figure 3.

Rotations are applied in the following order:

(1) rotation of $\varphi 1$ about Z-axis, (2) rotation of Φ about X-axis, and (3) rotation of $\varphi 2$ about Z-axis

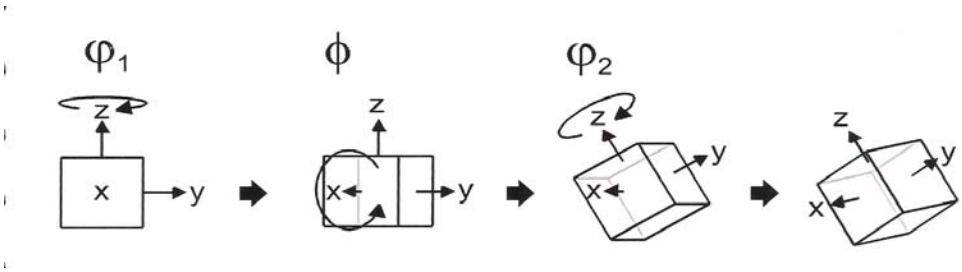


Figure 3. Standard rotation sequence for the determination of the Euler Angles, φ1, φ and Φ.

A second method for data representation is to maps using the crystal orientations (i.e. Euler angles) to create a pseudo color microstructural image. This microstructural imaging based on orientation has lead to this method being referred to as Orientation Imaging Microscopy (OIM). The OIM software will assign a particular color to each crystal based on the orientation of the cubic structure of the crystal at each point relative to the surface normal as shown in Figure 3. The color selected corresponds to the crystal orientation given in the standard crystallographic triangle as shown in Figure 4. This figure is related to the cubic structure of the atomic arrangements. Note that the vertex labeled <111> is the vector notation for the direction normal to the 111 plane and represents the cube diagonal. The cube face is represented by the notation <001> and <101> corresponds to the face diagonal. Therefore each color is assigned based not only on how it is physically related to the surface of the metal but to underlying atomic arrangement of the cubic lattice. In addition to this qualitative visual mapping of the crystal pattern of the sample, a quantitative evaluation of angular misorientations between adjacent crystals relative to the physical world or relative to any particular crystal within the structure can be calculated.

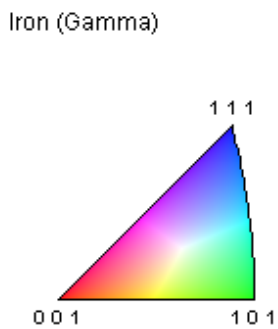


Figure 4. Standard crystallographic triangle relating grain orientation color to the vector normal of the crystallographic planes.

MATERIALS AND METHODS

This study examined metal fractures for two Fe-based alloys, a 304 (standard stainless steel) and an iron-gallium alloy (model system for orientational analysis). For the sample size there are several hundreds of crystals to measure assuming an average crystal size of 20 µm. The study examined known fracture matches to test the orientation analysis. Sample areas for investigation are selected based on examining fracture features, scale direction and size features.

304 Stainless Steel

For this work, a 304 stainless steel was examined because its microstructure is single phase, easily annealed, and is a base material for knives. The 304 stainless steel was cut into 12.5x12.5x3 mm coupons and annealed at 1050°C for one hour in vacuum. The annealing was performed to recrystallize the material and obtain randomly oriented, equiaxed grains. The coupons were mechanically polished using standard metallographic techniques and as a final step given a colloidal silica treatment for eighteen hours. Three scans were performed on three different 304 stainless steel coupons.

Iron-gallium alloy

Iron-gallium alloys rolled sheet samples were first fine ground and polished mechanically to a 0.05 um finish and then finished with a fine colloidal silica polish. The samples were notched on both sides in the center section of the specimen. The samples were fractured with force applied to the edge of one half of the thin sheet below the notched section. The two fracture halves were placed on a pedestal for examination in the EBSD. Orientation imaging microscopy (OIM) was used to produce orientation maps over an area of several hundred square microns. Individual orientations at locations were automatically measured using EBSD on a grid with a 2 um step size.

SEM and EBSD analysis

All scans were performed using standard EBSD operation with TSL OIM version 4.01 hardware and software attached to an Amray 1845 field emission gun SEM. Misorientation angles were calculated for each point along point-to-origin vectors applied to an inverse pole figure map (refer to Figure 5) generated by EBSD data. The misorientation angle between each location along the vector was calculated in reference to the origin. A grid of vectors with origins at a common point traveling radially outward at 5° angular separation was used. This grid was designed to have point-to-origin vectors of sufficient length needed to differentiate two adjacent vectors as a function of the grain size. To determine this length and divergent angles for these vectors, a simplified microstructure as illustrated in the schematic shown in Figure 6 using rectangular grains uniform in size and aligned, was used. The length was then calculated using the relationship: $L=GD/\tan(a)$, where a is the angle of separation, GD is the average grain diameter, and L is the vector length. Based on these calculations, a minimum vector length of 11.5 times the average grain diameter at a divergent angle of 5° was determined. This arrangement was used to hypothesize that each vector will result in a unique set of crystallographic orientations along its length relative to any other vector.

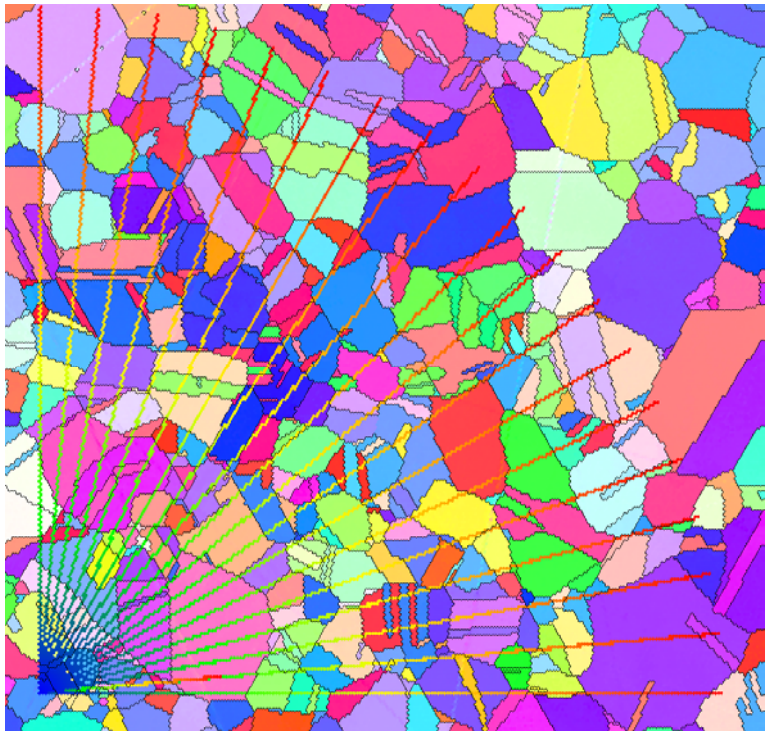


Figure 5. Grid placement for point-to-origin misorientation vector with 5° angular separation between vectors.

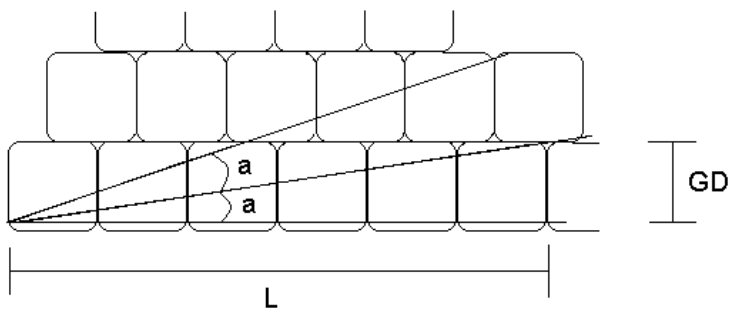


Figure 6. Schematic of grid placement for misorientation profile grid. *a* is the angular separation between point-to-origin vectors, *GD* is the average grain diameter, and *L* is the vector length.

The relative referencing method was used for grain sequencing [2]. This method compares angular misorientation values along a point-to-origin vector and when an angular difference of more than five degrees is found, it was reasoned that this was where one grain ends and another begins. The number of diffraction pattern data points in each grain is dependent on the step size and the distance through which the vector travels through the grain. Therefore, each misorientation value for points within a grain was averaged and an overall misorientation was assigned to that grain. The overall misorientation angle was then used in relation to its radial position from the origin; thus, creating a sequence of grain misorientations used for comparative analysis.

RESULTS AND DISCUSSION

304 Stainless Samples

EBSD scans were measured on three 304 stainless steel samples. A typical EBSD scan indicated an average grain diameter of 1.46µm and an average confidence index of 0.60. An inverse pole figure orientation map is shown in Figure 7. The distribution of misorientations between grains is shown correlated and uncorrelated in Figure 8. Correlated data consists of misorientations between just a grain and its next nearest neighbors, while uncorrelated data consists of misorientations between a grain and all other grains in the entire scan [3- 6]. For comparison of fracture surfaces, the correlated data is more applicable. To compare the fracture grain misorientation values with the rest of the sample grains, the uncorrelated data is useful.

The overall crystallographic nature of the microstructure was not found to be perfectly random. This is expected because it is nearly impossible to achieve a perfectly random texture [5]. Analysis of the predominate crystallographic orientations of the grains indicate there is preferred (111) nature to the microstructure as shown in Figures 7 and 8. However, the misorientation distribution is random enough for the purposes of this work. The uncorrelated misorientation distributions for two different scans shown in Figures 9 and 10 indicate a Mackenzie distribution for randomly oriented cubes. The average misorientations for the two scans are 40.42 and 40.63, very close to the ideal Mackenzie average of 40.7 . In the correlated data large peaks for high angle misorientation represent a high degree of twinning caused by annealing [4].

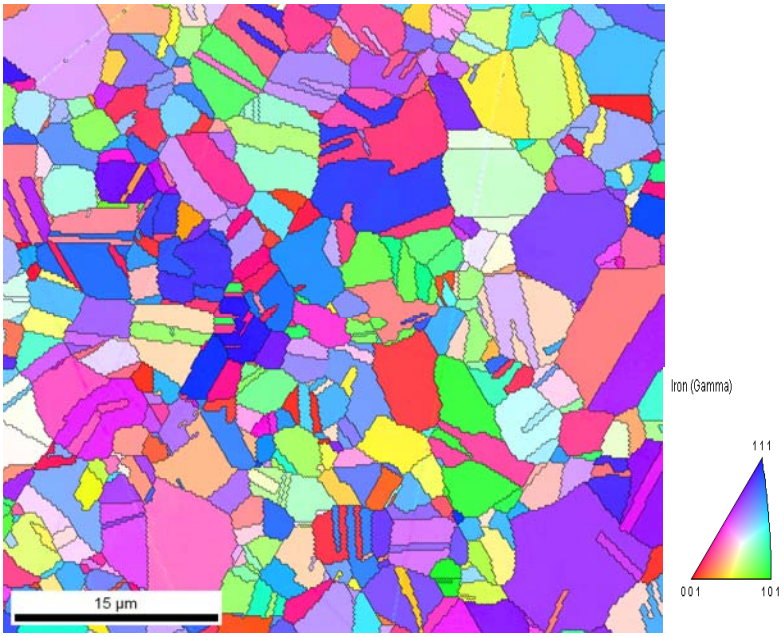


Figure 7. Inverse Pole Figure (IPF) map used for gathering misorientation data. Radial grid was applied and vectors were compared. Marker in lower left corner denotes 15µm. The small triangle is a standard crystallographic triangle relating grain orientation color to the vector normal of the crystallographic planes.

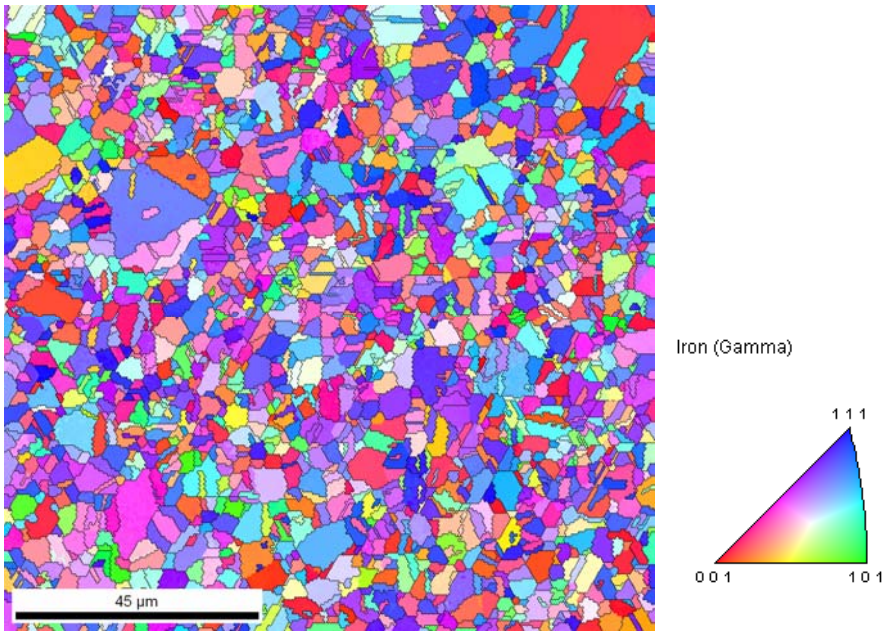


Figure 8. IPF map. Bar in the lower left corner denotes 45µm

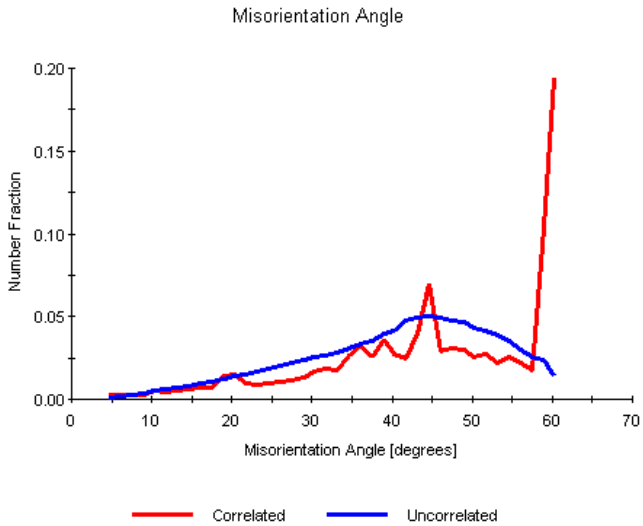


Figure 9. The uncorrelated plot closely resembles the Mackenzie plot with an average value of 40.42. The sharp increase at the end of the correlated data is due to annealing twins.

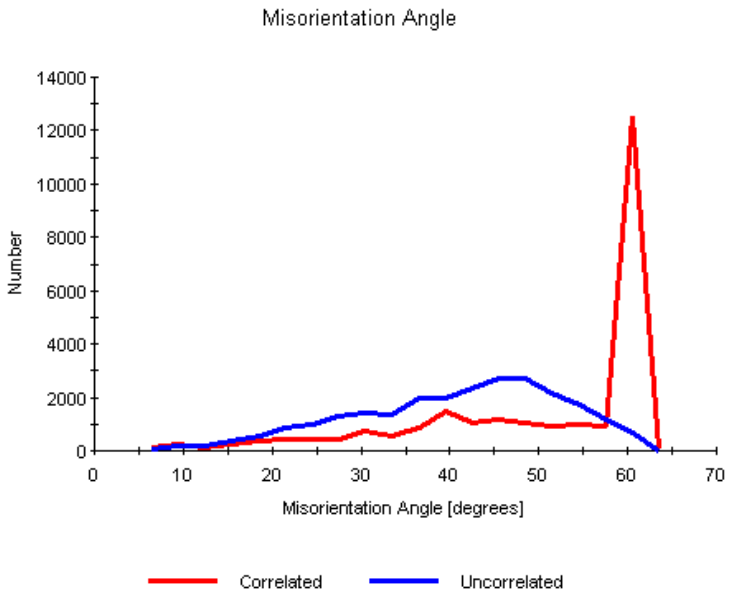


Figure 10. Average misorientation value is 40.63. Uncorrelated data shows close relation to Mackenzie distribution for random cubic misorientations.

A simple point by point matching parameter was applied to the point-to-origin misorientation vector output data. A vector was compared point by point with an adjacent vector with 5, 10, and 15° separation. Two points were determined to be in the same grain if the difference in orientation was less than five degrees of each other. The points were assigned a value of 1 if they were in the same grain, and given a value of 0 if they differed by more than five degrees. This grid was then used to determine how the sequence matching changed as a function of distance away from the origin and increased angular separation between vectors. A downward trend with increasing distance from the origin and increasing angular separation can be seen in Figure 11.

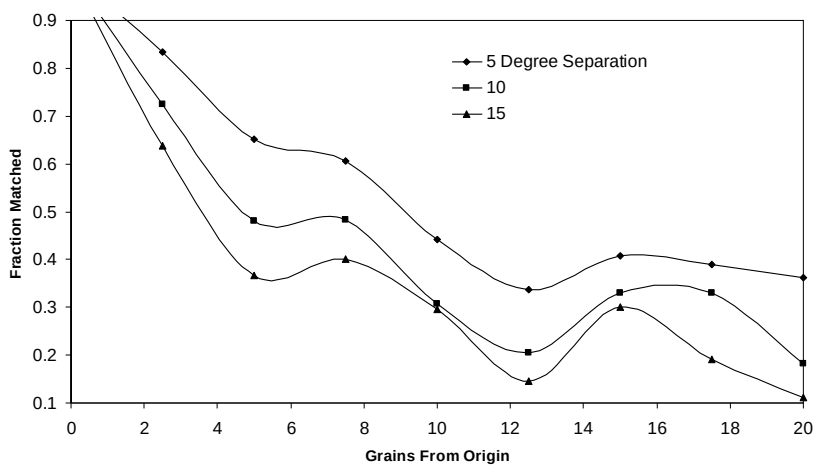


Figure 11. Plot of point to point matching data from all scans. The matching fraction decreases with distance from the origin vector and with increased angular separation.

The point-to-origin vector grid was placed on a common origin for each coupon of 304 stainless steel in order to test the hypothesis that geometrically, if a misorientation profile was taken over a certain distance of grains, the sequence of misorientations would be unique. Of the sixty-nine vectors applied to the EBSD data, no two vectors had identical misorientation sequences, i.e. no set of grains along any vector were identical to any other vector. In the sixty-nine vectors applied to the three separate scans of different areas, the average vector matched 2.6±2 grains with the adjacent vectors, with the longest match being nine grains. On average, each point-to-origin vector intersected 16.9±7 grains, more than the 11.5 grains predicted geometrically. An example of orientation sequences for microstructure in Figure 10 is shown in Table I.

Table I. Example of grain sequences from scan 3. The 0° and 5° vectors match two grains from the origin, while the 10° and 15° vectors match four grains.

	Grain Number													
Angle of Rotation	1	2	3	4	5	6	7	8	9	10	11	12	13	14
0	59.9	53.5	41.4	15.3	49.7	30.6	49.6	30.6	37.2	47.3	53.9	27.4	48.4	34.4
5	59.9	53.0	20.7	41.4	15.3	49.7	15.3	49.6	37.3	26.9	37.2	47.3	53.9	47.5
10	59.9	53.0	20.8	42.5	33.4	41.5	33.4	47.1	37.3	47.1	37.4	26.9	37.2	48.0
15	59.9	52.9	20.8	45.4	53.6	47.1	37.2	47.1	37.2	26.9	38.0	48.2	38.2	49.4

In this study, a simplified idealization of grains was assumed in order to estimate length and divergent angle values for the vector grid. In particular, it was found experimentally the number of grains analyzed along a given vector was greater than the number predicted by the simplified model. This is because in reality grains are non-uniform in shape and can be present in a wide range of sizes and morphologies as seen in Figure 5. The large variability in the number of grains characterized per vector can be attributed to various sizes in grains and vectors not traveling through the center of each grain. Therefore each vector contained more grain-to-grain misorientations for comparison than originally anticipated and therefore can be more easily distinguished from its adjacent vectors.

In the point by point matching parameter, vectors placed 5° apart had 83±11% of data points match for the first 2.5 grains away from the origin. In the next 2.5 grains, the percentage dropped to 65±5% matching points, and in the last 2.5 grains 36±10% of data points match. This shows that through grain reconstruction it is possible to demonstrate that similar or non-similar orientation points are being indexed. In comparing the misorientation sequences along adjacent vectors starting from the origin, the number of grains that were similarly misoriented with respect to the origin grain was found to be 2.6±2 grains. Based on these results, a 5° divergent angle provides adequate resolution between adjacent vectors to correctly describe the misorientation sequence, even if the vectors are not perfectly aligned as might be the case when analyzing two pieces of metal across an alleged fracture pair.

Iron – Gallium Alloy Samples

The OIM orientation map generated by the EBSD scans of the fractured iron-gallium alloy sample is shown in Figures 12 and 13. The figures are showing the orientation of the grains along the fracture edge. The particular color for each crystal is based on the orientation of the cubic structure of the crystal at each point relative to the surface normal. Matching colors across the fracture edge along the path indicate a transgranular path. Areas where there are different colors across the fracture indicate portions of the fracture path where fracture occurs on the boundary between grains. Figure

13 is the same fracture shown in Figure 12 but this time the color scheme is assigned using Euler angles. The Euler orientation angles for pink, purple and brown grain orientation colors are given in Table 2. This table shows an example of the type of match between two fracture edges that is *good* considering that fracture along the edge included a path between some grains that are not present in both the top and bottom. The fracture was intragranular (between grains) at certain locations.
Figure 14 shows a higher magnification of a section of the fracture from Figure 13. For the Fe-Ga alloy clear one-to-one matching of the crystals across the fracture surface is possible using either crystallographic information or misorientation angles (Euler angles).

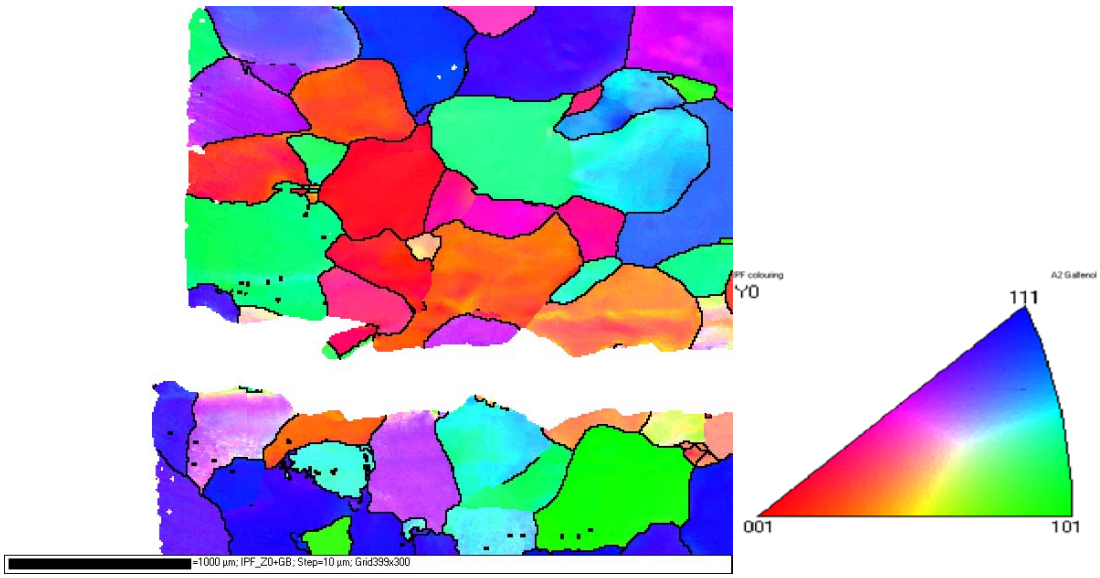


Figure 12 Orientation imaging microscopy maps of the top and bottom of the steel sample including the fracture edge.

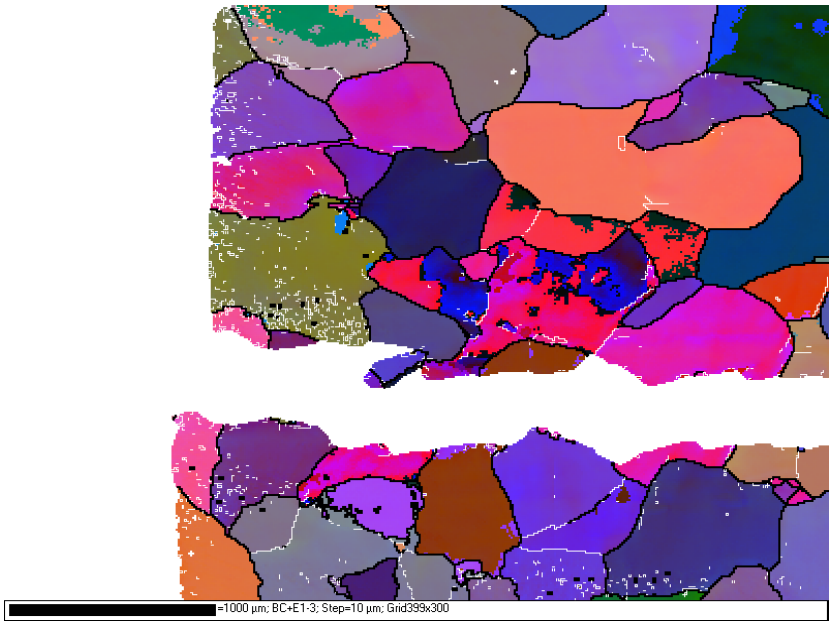


Figure 13. Orientation imaging microscopy maps of the top and bottom of the steel sample including the fracture edge.

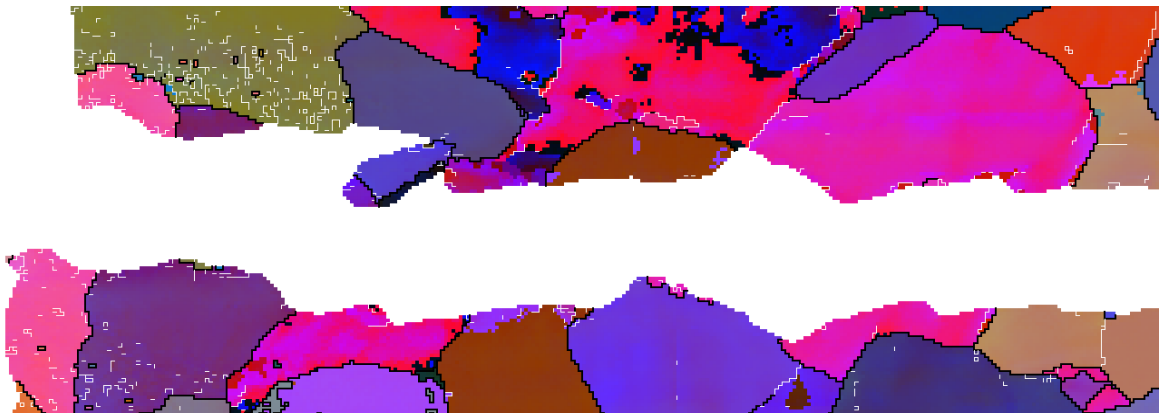


Figure 14. Orientation imaging microscopy maps of the fracture edge of the top and bottom of the steel sample.

TABLE 2 Three Orientation Euler Angles For Grain parts on two sides of fracture.

Edge	Color	Angle Φ1	Angle Φ	Angle Φ2
Bottom	Pink	341°	29°	56°
	Purple	162°	16°	47°
	Brown	202°	19°	3°
Top	Pink	343°	28°	53°
	Purple	169°	11°	38°
	Brown	198°	19°	6°

Uniqueness of linear sequences

The profile of grain to grain misorientations along a length can be used to distinguish fracture surfaces. A detailed statistical treatment of the data would be needed to exactly determine the number of grain orientations necessary to uniquely identify a fracture line and is beyond the scope of this project (see Appendix for additional details of this effort). A detailed non-standard statistical approach is currently being developed as part of another study. Here we provide a simple estimate of the probability of any particular sequence, assuming the (mis) orientations of grains are random relative to one another within a metal. For the general case, a sequence of grains with each grain having X possible orientations, yields a probability of any particular sequence of orientations

$(1/X)^n$ where n is the number of grains in the sequence.

Here X is determined both by the symmetry of the system as well as by the resolution of the measurement method In cubic rotations there are twenty-four angles of rotation which will bring cube B into coincidence with cube A and twenty-four angles of rotation which will bring cube A into coincidence with cube B. This gives 576 rotations, and factoring in improper rotations (inverse direction of a proper rotation), this gives 1152 axis-angle pairs that can be used to describe a single misorientation value [7]. Even with this large number of axis-angle pairs to describe one misorientation state, the resolution of EBSD techniques allows for an even greater number of distinct

crystal orientations. With an angular resolution of 4°, 130,000 distinct orientations are detectable and with an angular resolution of 2°, the figure increases to 4,155,000 [8].

For an angular resolution of 4 degrees, there are more than 130,000 distinct possible orientations for each of the grains along the sequence of grain orientation values. [8]

$(1/130{,}000)^n$ where n is the number of grains in the sequence.

For a sequence of six grains, assuming that all grains have the same possible number of potential distinct orientations, the resulting probability that the same sequence of six grains will occur again is 2^{-31}.

SUMMARY AND CONCLUSIONS

The purpose of this study was to evaluate whether surface crystal orientation can be used to associate metal fracture fragments. This study sought to test if the orientations of the fractured crystals across the fracture plane for two surfaces determined by a forensic examiner to be a matching fracture by conventional methods. This study used Electron Back-Scattered Diffraction (EBSD), sometimes known as Orientation Imaging Microscopy (OIM), to determine the crystallographic orientation of individual metal crystals along the length of fracture surface on a surface normal to the actual fracture. This study showed that in the case of intragranular fracture, pieces of grains could be associated across the fracture surface. This study showed it is feasible to associate two fracture pieces with sequence of grain orientations. It is also possible to use sequence of the difference in orientation between grains (misorientation) along a length on one side of the fracture surface to associate the other side of a fracture surface.

Orientation angles for grains and misorientation values between grains were calculated and the resulting values were compared for grains across fracture surfaces. For iron gallium samples, it was shown that it is feasible to use surface crystal orientation to associate one fracture edge to another. Regarding the uniqueness of crystal orientations within a metal, it was estimated that for a sequence of six randomly oriented grains along a fracture, the resulting probability that the same sequence would occur again is 2^{-31}.

In this work the grains were sequenced exclusively by their relative crystallographic orientations. This approach limits characterization to only one of several microstructural features than could be used as a unique identifier. Another parameter which has potential for being used in combination with orientation is grain size and shape along the fracture line. This is a facet which should be further examined in future work.

DISSEMINATION

Results of the feasibility study were presented at the Midwest Forensics Resource Center Annual Meeting in Madison, Wisconsin in April 2007. A poster of the results was presented and discussions were conducted with forensic professionals at the IAI Annual meeting in San Diego, CA in July 2007.

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APPENDIX

Preliminary Statistical Evaluation of the EBSD technique.

Dr. Barbara K. Lograsso
Ames Laboratory
120 Spedding Hall
Ames, IA 50011
blogr@ameslab.gov

Phone :(515) 294-2931
Email: blogr@ameslab.gov

Dr. Thomas A. Lograsso
Ames Laboratory
111 Metals Development
Ames, IA 50011
lograsso@ameslab.gov

Phone: (515) 294-8425
Email: lograsso@ameslab.gov

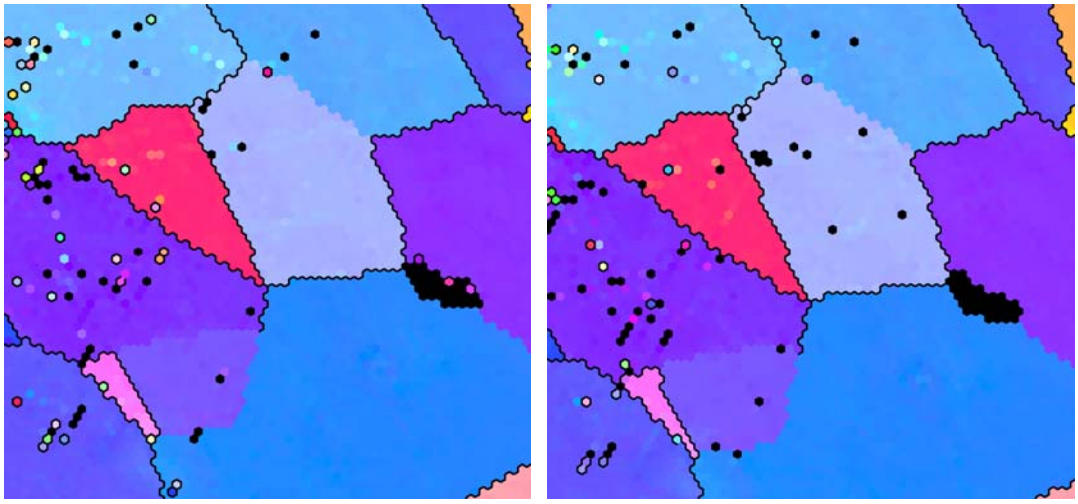
Dr. Dan Nordman
Iowa State University
Department of Statistics
315F Snedecor Hall
Ames, IA 50011
Email: dnordman@iastate.edu

Dr. Stephen Vardeman
Iowa State University
Department of Statistics
304D Snedecor Hall
Ames, IA 50011
Email: vardeman@iastate.edu

Melissa Bingham
Iowa State University
Department of Statistics
315E Snedecor Hall
Ames, IA 50011
Email: melissa@iastate.edu

Analysis of the technique has been focused on statistical examination of the ESBD method applied to crystal orientation. This examination has begun with an assessment of the variance and measurement error for the technique. This analysis involves measuring the same area multiple times with at least 4000 measurements per run on the sample surface area of size 40µm x 40 µm. These tests are initially being conducted on TSL (EBSD company) calibration standard Iron-high concentration nickel sample, which shows ideal EBSD patterns.

Figure A1 shows a map of the standard's microstructure. The surface of the sample was prepared to give favorable EBSD patterns. 4000 EBSD patterns were taken at intervals over the field of view and the resultant orientation patterns were indexed to the crystal structure of the TSL sample (generally cubic in nature for most metals).



Figures A1a and A1b. Backscattered electron orientation patterns are collected from the same positions on the TSL standard sample surface.

The above images show the repetitive examination of the same area on a TSL (EBSD company) calibration standard Iron-nickel specimen and demonstrate the high degree of repeatability of the technique to determine the underlying crystal orientation. The patterns shown above are representative of a larger collection of data generated for statistical analyses. One can notice in the regions of good strong patterns, indicated by solid coloring and clear lines denoting changes in orientations between crystals, the images are identical. However, in certain regions, for instance, the dark spot on the grain boundary, the assignment of orientation from the EBSD patterns were either nonexistent or weak. This failure to adequately determine orientations may be of significance when analyzing materials and specimens that do not exhibit strong EBSD patterns.
Because the performance of any measuring equipment used in a study must be known to be adequate, a statistical analysis is being done to study the repeatability of the process used to determine the underlying crystal orientation. The same specimen was measured 14 times, giving close to 4000 samples of size 14. A statistical model was formulated to study the error within each of these samples. The model consists of uniformly selecting a point on a sphere and rotating the true location of the crystal axes around this point at an angle of size θ, where θ comes from the Wrapped Normal distribution on the circle. Figure A2 shows the density of the Wrapped Normal distribution, with mean 0, for various values of the standard deviation. This figure illustrates the difference in spread for various choices of standard deviation.

For each sample of size 14, maximum likelihood estimation was used to give an estimate of the true orientation of the crystal axes and to give an estimate of the standard deviation of the Wrapped Normal distribution. The standard deviation essentially measures the error within a sample. A small standard deviation gives a distribution with little spread and is evidence of the high repeatability of the measurement process. Figure A3 is a grayscale map of the surface that was measured, coded by the estimated standard deviation of the Wrapped Normal distribution.

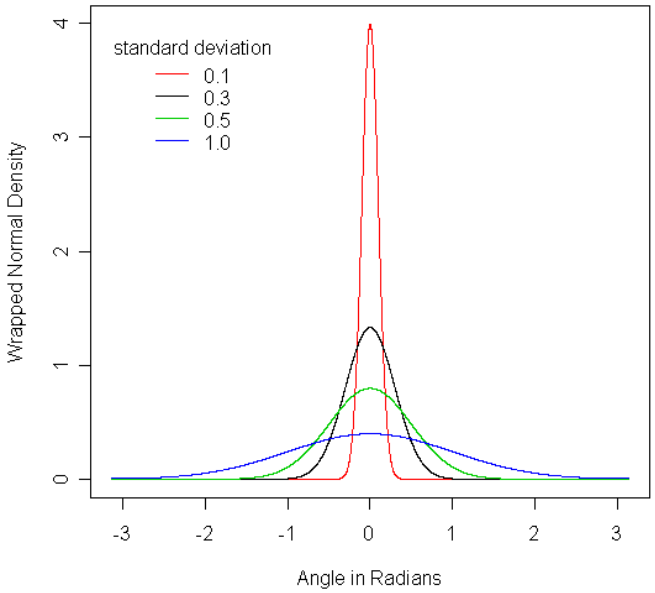


Figure A2. Density of the Wrapped Normal distribution for various values of the standard deviation

The points on the map in red indicate a place where at least one of the 14 observations was a missing value. One can see that the majority of the points are white, indicating a very small standard deviation within that sample. This is overall evidence of the high repeatability of the measurement process. When comparing Figure A3 to the earlier colored figures, one can see that the points that are a darker shade of gray appear to lie mostly along grain boundaries, likely due to the fact that it is harder to get accurate measurements along these boundaries.

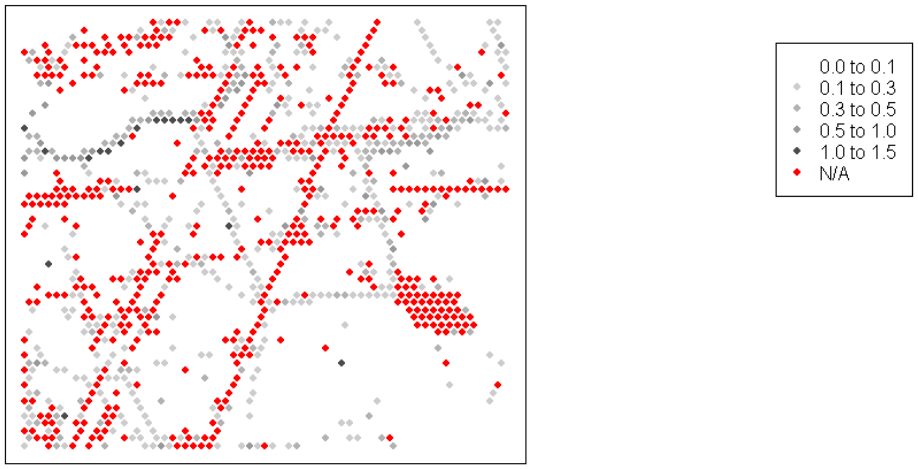


Figure A3. Plot of the measured surface coded according to the standard deviation among measurements

Future statistical analyses will include an assessment of the model that has been used to study this repeatability. It is important to know how accurate the measurements obtained from the maximum likelihood estimation are. This can be studied by generating many large samples from the proposed model, in which true values are set for the parameters. The estimation is then done on these simulated samples to investigate the amount of error present in the estimation.

Recently, the model developed to study the repeatability of the measurement process was modified slightly. The model still consists of uniformly selecting a point on the sphere and rotating the true location of the crystal axes around this point at an angle of size θ, where θ comes from a circular distribution. Instead of using the Wrapped Normal distribution as our circular distribution, we are now using the Von-Mises distribution, which has a more simplified density. The representation of the likelihood equation was also modified, giving a much smoother curve. The smoothness of the curve allows us to more easily and accurately obtain maximum likelihood estimates for the parameters of the model, which are the true orientation of the crystal axes and the standard deviation of the Von-Mises distribution.

Now that we feel we have an adequate model for studying repeatability, we have begun to work out the statistical asymptotic theory behind this model. This will allow us to derive confidence bounds for the parameters in the model, test for specific values of these parameters, and assess the fit of the model to the data at hand.