Reducing Texture Effects on Austenite Phase Fraction Calculations with X-Ray Diffraction Techniques

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Abstract

X-Ray Diffraction (XRD) is one of multiple industrially-relevant methods used to measure phase fractions in formed steels. However, this method can be prone to rather extreme measurement bias. Crystallographic Texture in particular is a phenomenon known to be a source of these measurement errors. This project, which specifically focuses on measuring retained austenite in steel samples, seeks to implement and test new sampling methods which can help reduce this bias. For the rather limited timeframe and scope of this project, I will be focusing on the Equal Angle Scheme. My job will be create a function for this scheme through code, run simulations with this function, and code for a data visualization tool which will allow me to process all the phase fraction values at once as a function of several texture components. With the help of this heatmap, I will be able to easily see which texture components continue to be problematic for the equal angle scheme (in terms of continued high measurement bias) and which texture components are taken care of by the scheme (in terms of measuring the phase fraction with relative precision and accuracy). As formed steel products often implement retained austenite to enhance the strength and ductility of the steel sample, it's imperative for users and producers of steel alike to know how much retained austenite is in their steels to appropriately predict its mechanical response.

1 Texture: An Introduction

As visualized in **Figure 1**, a round-robin study conducted by *Jacques et. al* [1] demonstrates the incredibe discrepancies in retained austenite phase fraction measurement. The suspected culprit for these inconsistent measurements is of crystallographic texture, which is a preferred orientation of grains within a crystal lattice. When grains are in completely random orientation, we say that there's no texture present, and vice versa.

Though its exact origins and formation are still a topic of research, it's known that texture can manifest itself during the mechanical processing stage of the steel. In particular, we investigate bar and sheet steels, which are formed by taking steel samples and rolling them uniaxially, thereby 'flattening' them into steel sheets. In this process, it's not uncommon to notice a common orientation of grains along the 'rolling axis'if we were to investigate the microstructure of the sheet sample.

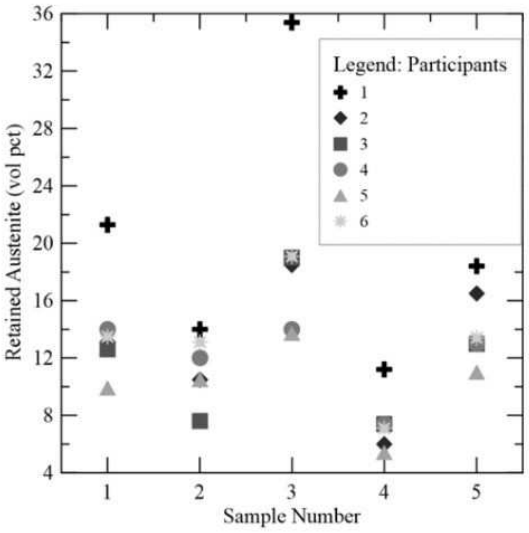

Figure 1. A graph representing the incredible discrepancies in phase fraction measurements of retained austenite in steel samples, by 6 different members.

The fundamental formula that governs X-Ray Diffraction is the Bragg Condition and Formula:

$$n\lambda = 2d\sin\theta \tag{1}$$

Where n is an integer value, λ refers to the wavelength of the X-ray particles, d refers to the slit distance between adjacent grains in the crystal lattice, and θ refers to the angle of impact of the X-ray beam.

In an ideal case, XRD scan results are represented in a series of graphical 'peaks' (**Figure 2**) indicating the existence of a certain phase. It is important to realize that the height of each diffraction peak doesn't correlate with more of the phase present in the sample. This only means that the relevant angle measurement was more cleanly diffracted and well-received by the scan detector (constructive interference of outgoing X-ray beams). These 'peak' intensity values, which are more organized by their planar location in the atom, are all then aggre-

gated appropriately to solve for the phase fraction value. However, when texture is present, it can impact the intensity values of these peaks by shifting them along the graph, thus affecting our calculations of retained austenite present in the sample.

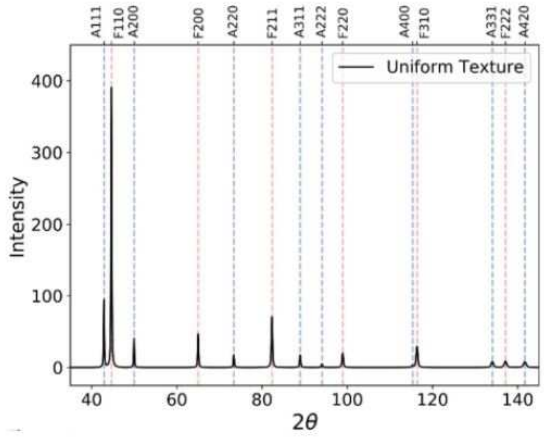

Figure 2. A sample, generic XRD scan output. The 'peaks' of the graph indicate the existence of a certain phase at a certain orientation.

Texture is a very broad and overarching concept which can manifest in multiple different ways. For particular commonly-occuring texture mechanisms, there are proper names that have been assigned to them (Goss, Shear, etc.) These are called 'Texture Components.'This project only explores a handful of these commonly-occuring texture components, so when particular names are referred to later on in this report, please know that they refer to these well-documented texture components.

2 Backgrounds and Methodology

Avoiding the mechanical formation of texture is virtually impossible, so this project focuses on developing new sampling methods which can mitigate these negative effects of texture. In order to develop and test sampling methods that can effectively reduce measurement bias, my mentors turned to computational procedures. Through python scripts, they prototyped various promising 'sampling schemes,' or specific orientations of sampling points of the XRD scan [2]. For example, a 'hexagonal scheme' gets its name from the sampling points being arranged in the rough shape of a hexagon, and so on.

The X-Ray Diffraction scan setup is inherently a 3-Dimensional process, and it can be difficult to visualize the orientation of various XRD sampling points in a 3D setting. Sampling points within an XRD scan are usually parametrized by the angles of measurement about each of the X, Y, and Z axes. In this project, we use pole figures to represent these collection of sampling points on a 2 Dimensional plane. To better illustrate this concept, an example of the pole figure for the equal-angle scheme is shown in **Figure 3**, where each of the axes are represented by ND (the 'Normal' Direction – the one pointing straight up towards the ceiling), RD (the 'Rolling' Direction – running parallel to the counter or surface where the sample is kept) and TD (the 'Transverse Direction' – the axis which lies on the plane of the table (running parallel to it) and also runs perpendicular to the RD axis.) Such stereographic projections have simplified the way we visualize and develop sampling schemes, and are the basis for how we choose to name certain sampling methods.

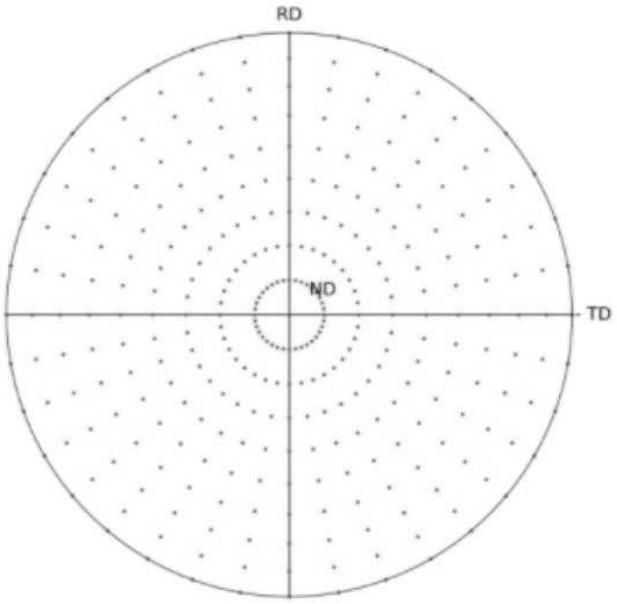

Figure 3. A stereographic output for the Equal Angle scheme. Oversampling is apparent towards the center of this pole figure; undersampling towards the outside. The different 'axes' have also been labeled (RD, ND, TD).

With the help of Python's 'mplstereonet' package, we can visualize our schemes after coding for them in a function which arranges these sampling points in a given manner. This is used to make sure we coded the scheme correctly, and predictions can be made as to how well we believe the sampling scheme will perform based on how well the sampling scheme 'covers' the projection grid. For my 'Equal-Angle' sampling scheme, you can see how there is oversampling towards the center of the projection grid; conversely, undersampling exists as we move farther and farther away from the center. This is inherent to the code itself; although methods exist to account for such over and undersampling by selective 'weighting' of sampling points, that is completely beyond the scope of both this project and my knowledge as a mere high school student. What we do know however, is that this sampling scheme is predicted to have significant issues when it comes to mitigating the effects of certain texture components. Which texture components this scheme will struggle with is the specific focus of this project, as covered in the 'results' section.

After coding for the Equal Angle scheme, the next step involves plugging these into a simulation, which had been developed by my mentors beforehand. This simulation numerically mimics the X-Ray Diffraction process by outputting simulated intensity values as a function of various parameters (such as

scheme chosen, number of peaks chosen to incorporate into the phase fraction calculation, etc.) It is important to take note of the number of peaks the simulation will account for in the calculation of phase fraction. While this is another independent variable I will be modifying in my SRP, I decided to limit the scope of this project by setting the number of peaks to the generally-accepted value of 3. As seen back in **Figure 2** on this page, peaks of higher intensity values can be seen at certain orientations of the sample. This is important to consider because certain texture components are aligned in unique orientations within the sample. Depending on the scanning orientation, whether or not the texture component runs parallel or perpendicular to the direction of measurement can impact the ability for the scan to accurately measure phase fractions within the steel. Again, this is an idea that wasn't explicitly focused on in this project, but can be a source of explanation for why certain texture components appear more or less problematic for a given scheme. Finally, the 'actual' or real value of the phase fraction is the final parameter that can be passed in as well, and for this project, I went with a standard retained austenite phase fraction value of 0.25. Again, when testing steel samples for this project, most metallurgists either go with samples with a known composition of retained austenite (by volume) of either 0.25 or 0.54. For this project, I decided to go with 0.25, as it is a more standardly-used composition for these applications.

Finally, I developed a 'Heatmap' function, which, given a sampling scheme and peak combination used for phase fraction calculation, outputs a heatmap to visualize all the relevant phase fraction values. The heatmap has been appropriately grayscaled to allow for use in professional reports. It sorts through hundreds of intensity files (outputted from the simulation) and consolidates the relevant data into an easy-to-understand format for viewers. Perhaps most importantly, each cell of the heatmap table corresponds to a 'combination' of 2 texture components. This allows users to easily determine which combinations of texture components appear most (or less) problematic given a certain sampling scheme.

To clear any potential confusion, the viewer will notice the axes of the heatmap labeled 'Austenite Components' and 'Ferrite Components.' Ferrite is another phase present in microstructure, and is one commonly-occuring in the steel processing stages. Naturally, textures can appear in the ferrite phase as observed in the steel's microstructure; however, I didn't explicitly go into detail on discussing Ferrite because it is not the phase of our focus here. Retained austenite is the phase we are more interested in, but we must still take into account naturally occuring textures that exist in the Ferrite phase of a steel's microstructure as this will nonetheless impact our calculations.

3 Results

Figure 4 near the top right of this page summarizes the results of the performance of the Equal-Angle scheme. According to our heatmap, it's clear that the 'Shear' Ferrite component and the 'Cube' Austenite component seem to be the two texture components that are most problematic for the Equal Angle sampling scheme. For a known volume phase fraction of 0.25, the 'Shear' texture component consistently underestimates the phase fraction value, while the 'Cube' texture component consistently overestimates the phase fraction value.

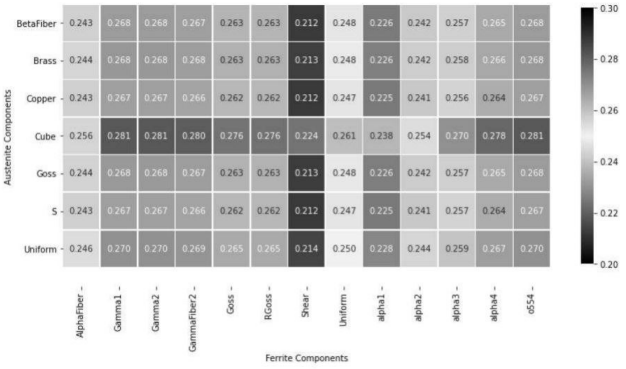

Figure 4. A Heatmap displaying Phase Fraction Calculations with the Equal-Angle Scheme. The Shear Ferrite and Cube Austenite components stand out as texture components which the Equal-Angle scheme struggles with in particularl.

As an important side note, this heatmap includes 'uniform' textures for both ferrite and austenite texture components. These both serve as reference fields: noticing the intersection of 'Uniform' ferrite and 'Uniform' austenite to hold a phase fraction of 0.25 confirms this idea.

A feasible explanation for this phenomena ultimately lies beyond the scope of this project and requires a deep analysis of texture components and their orientations in microstructure. This is something I want to briefly explore within my SRP, though it is admittedly a bit ambitious. One hypothesis I would like to test involves the orthogonality of the texture component vector and the direction of measurement. If the two vectors are orthogonal, I would predict that the effects of texture will be greatly exacerbated, and if the direction of texture component runs parallel to the sampling direction, the effects of texture will be better mitigated. Again, this is most influenced by which 'peaks' and therefore which sampling orientations we choose to incorporate in our calculation, which is another parameter I plan to focus on more in my SRP.

As I code for multiple schemes and process the results, I will observe whether the 'Shear' and 'Cube' texture components consistently act up regardless of the scheme. This could indicate the extent to which the directions of texture component vectors impact the ability for the XRD scan to accurately measure the amount of retained austenite.

At the end of the day, it's a balance of practicality and performance. Although we observe upto 10-12% deviation in phase fraction measurements for the equal angle sampling scheme, it also holds some promising results. The scheme effectively reduces bias caused by the 'AlphaFiber' ferrite texture component, though it does not consistently perform as well for any of the austenite texture components. Compared to other proposed sampling schemes, the equal angle scheme is easier to carry out in actual XRD scans and less computationally tax-

ing. Should researchers know in advance that a steel sample to be measured for retained austenite is textured only with an 'AlphaFiber' ferrite component, the equal angle scheme could be an option for them. In my SRP, however, I hope to reveal more promising sampling schemes which combine versatile and consistent performance with equally impressive feasibility in the lab room.

4 External References

The following section enumerates two sources that were not explicitly called in the above texts. However, I referenced them nonetheless for general information needed to write this report. [3] was my main general reference throughout this entire process, as it provided a very general outline of materials science and engineering for me to contextualize this project in. [4] is a website I looked up on my own to better understand how X-Ray Diffraction is used to determine crystalline structure and composition of materials. Please find the references on the final page below.

References

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