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Comparison of grain size distributions in a Ni-based superalloy in three and two dimensions using the Saltykov method

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A three-dimensional (3-D) dataset of Ni-based superalloy Inconel 100 is used as a validation case for using stereology to estimate 3-D grain sizes from 2-D data. 2-D sections of the IN100 dataset are extracted, from which 3-D size distributions are estimated through the use of the Saltykov method and compared to the true 3-D statistics. The Saltykov method corrected the upper tail disparity between the 2-D and 3-D grain size distributions, but the lower tail of the distribution was not improved.

Keywords: Grain size distribution; Fatigue; Microstructure; Twinning; Superalloy

Emulating microstructure–property relationships requires simulations of the microstructure, and measured three-dimensional (3-D) datasets are rarely obtainable. The use of 2-D data to predict 3-D metrics can introduce significant inaccuracies. This is particularly true of extremes in grain sizes, for example, which can be significant for fatigue resistance. Applying the Saltykov method to grain sizes measured in 2-D sections produces a more accurate 3-D estimate.

Although it is possible to reconstruct the 3-D structure of polycrystals, the materials genome of 3-D datasets is not yet large enough to accommodate current needs [1,2]. Nor is it inclusive of datasets large enough to resolve the entire distribution of grain sizes (a general guideline is 10,000 grains). Additionally, the time and cost of collecting these datasets is often prohibitive. Significantly more grain data exists in the form of single or limited multiple orthogonal electron backscattered diffraction (EBSD) scans, and the grain sizes are usually only available as linear intercepts. Legacy data of this type was the standard for decades before the emergence of 3-D characterization techniques. Accordingly, the ability to estimate the 3-D distribution of grain sizes from 2-D data will always be valuable, and this is the focus of this communication.

Most studies have focused on applying Saltykov's analysis [3] to particle distributions [4–6]. Saltykov's analysis has been applied to distributions of grains [7], as proposed here, but without real grain data from a 3-D dataset to validate the process and without specific attention on large grains (often critical to microstructure–property relationships). Because it is commonly accepted that many single-phase fully dense polycrystals are described by a log-normal grain size distribution [8–10], this treatment of the Saltykov method is referred to as the Johnson–Saltykov method, which is applied on a logarithmic scale. The Johnson–Saltykov method is referred to as the Saltykov method in the remainder of this document [11].

The primary difference in examining particles vs. grains is the assumption of isolated particles (zero contiguity) as opposed to space-filling grains – an important point that will be addressed later. This study represents a synthesis of existing data and techniques that should be applicable to many types of legacy data, not just Ni-based superalloys. We validate the approach by applying the Saltykov method and using the existing 3-D IN100 dataset as a direct comparison. A significant benefit of these findings is that 3-D statistics can be accurately estimated even in the absence of true 3-D

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data, thus demonstrating the feasibility of the stereological reconstruction. This study also reveals differences in 2-D and 3-D grain data. Defining a microstructure as equiaxed does not guarantee that a single plane of data will be representative of the bulk material.

The acquisition of the IN100 dataset is explained in Ref. [1] and the process for twin removal is found in Ref. [13]. The resulting model is shown in Figure 1. Twins represent an additional element of complexity and are not considered in this study. After the twins were removed, the IN100 microstructure was voxelized on a cubic grid with a lattice spacing of 0.25 μm. The dataset is $398 \times 146 \times 184$ voxels and has 1818 bulk grains. Only bulk grains are considered in this study so that grains with incorrect volumes are not added to the distribution.[1] The software package DREAM.3-D was used to place the serial sectioned data on a grid and calculate the distribution of grain sizes [14]. DREAM.3-D is developed at the Air Force Research Laboratory (AFRL) with contributions from Carnegie Mellon University [15–17].

The Saltykov method [3] predicts grains per unit volume from grains per unit area. It does this by assuming all grains are spheres and making an adjustment based on the intersection probability of a sphere with a section plane. The linear intercept data is binned and an unfolding algorithm is applied as shown in Eq. (1):

$$N_{VK} = \frac{1}{D_K p_1}\left(N_{AK} - p_2 N_{B(K-1)} - p_3 N_{B(K-2)} - \cdots - p_K N_{B1}\right) \tag{1.1}$$

$$N_{BK} = D_K N_{VK} \tag{1.2}$$

Here, D_K represents the as large as (ALA) grain of the 2-D data. This procedure is described in greater detail elsewhere [4–7]. Proper selection of the number of bins is important so as to not over- or under-discretize the binning. In general, selecting a bin width equal to the grid spacing in the image produces a reasonable number of bins. Here, a bin width of 0.25 μm is chosen, which produced 56 size bins.

Previous work [16] has shown that it is reasonable to treat the grains in IN100 as equiaxed, which enables the use of a 2-D map to estimate 3-D grains. The linear intercept data is obtained by performing a line scan on the voxelized IN100 microstructure. The line scan was conducted by moving along the x-direction in the x–y plane and defining an intercept as when the grain identification changes. Once an edge is reached, the scan shifts one voxel along the y-direction before proceeding again along the x-direction. This is similar to the procedure for gathering linear intercept data from an EBSD scan or grain boundary map using standard analysis software. The number of intercepts used is comparable to the number of 3-D grains, so as to produce a similar number of estimated 3-D grains after the Saltykov method is applied. Also, edge intercepts are not included in the sample. The results of the line scan and application of Saltykov's analysis compared to the sphere equivalent 3-D grains are displayed in Table 1 and Figures 2 and 3. Figure 3 depicts a probability plot which represents how normal – or, in this case, log-normal – the data is. The datasets exhibit a mean field log-normality with different degrees of lower and upper tail departure [18].

Figure 1. Inverse pole figure of IN100 with twins removed. It comprises 1818 bulk grains and $398 \times 146 \times 184$ voxels, with 1 voxel = 0.25 μm.

In this data, a minimum threshold of 1 μm sphere equivalent or caliper diameter is enforced as a reasonable limit of feature resolution. Any grain below this limit is not included in the data analysis. From the samples used in this study, 513 intercepts from 3150 and 9 grains from 1818 grains were below the threshold size and eliminated (denoted as Data Cutoff in Table 1). First comparing the caliper diameter to the sphere equivalent diameter, in the upper tail, the linear intercept method on average will overestimate the ALA grain. Although IN100 is assumed equiaxed, the grain aspect ratios deviate from unity. If a linear intercept captures the major axis of a near-ALA ellipsoidal grain, it will overestimate any existing sphere equivalent diameter in the polycrystal. The result is a linear intercept upper tail that overestimates the sizes of the largest grains. The Saltykov method acts to correct this overestimate. The ALA grain sphere equivalent diameter (D_K in Eq. (1)) is 12.37 μm, compared to the Saltykov estimated 13.44 μm. This is greater than the 3-D data, but the upper tail of the linear intercept grain size distribution after the Saltykov method is applied more closely coincides with the 3-D data (Fig. 3).

Table 1. Comparing different diameter metrics of the IN100 linear intercept, the IN100 sphere equivalent and the Saltykov method performed on the IN100 linear intercept.

	IN100 Linear intercept	IN100 Sphere equivalent	Saltykov: IN100 linear intercept
Sample size	2627	1809	2105
Bins	56	50	56
Data cutoff	513	9	1
ASA (μm)	1	1	1
ALA (μm)	13.75	12.37	13.44
Mean (μm)	3.61	3.65	3.31
Mu (log(μm))	1.1	1.19	1.04
Sigma (log(μm))	0.61	0.46	0.56

[1] Note that this treatment still has a small bias towards small grains. An alternative unbiased method called "guard frame" is described in Refs. [2,12]. The use of a "guard frame" entails excluding grains whose center of mass is within an imposed frame between the domain edge and an inner boundary usually defined as restrictive enough to eliminate all features that touch the edge.

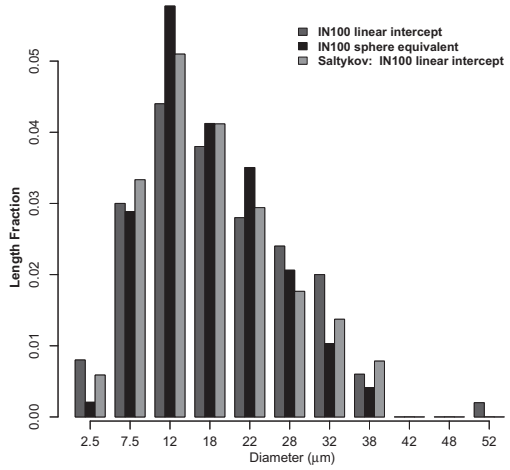

Figure 2. Histogram comparing the IN100 linear intercept, the IN100 sphere equivalent and the Saltykov method performed on the IN100 linear intercept. The Saltykov bins match the 3-D grain data bins more closely than the IN100 linear intercept bins.

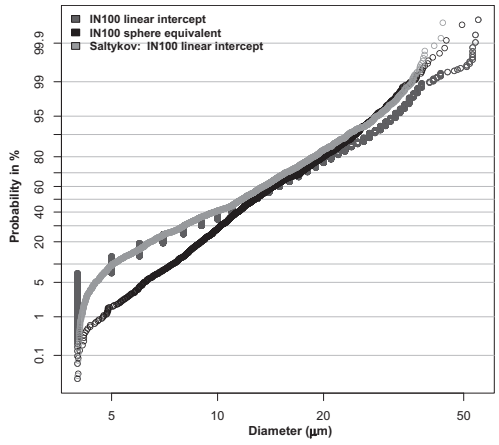

Figure 3. Probability plot comparing the IN100 linear intercept, the IN100 sphere equivalent and the Saltykov method performed on the IN100 linear intercept. The Saltykov method corrects the upper tail departure, but the lower tail disparity prevails. Note the the x-axis is plotted on a log scale.

In the lower tail region, the linear intercept data is voxel discretization, which is the limit of accuracy on a grid. This is more evident in the lower tail because of the logarithmic scale. The linear intercept method underestimates the 3-D grains in the lower tail; however, the Saltykov method does not correct this disparity. Two main sources are suggested to account for this error. Figure 4 portrays the aspect ratio. In Figure 4 there are many nonspherical smaller grains relative to the rest of the distribution of grain sizes. As a result, because the sphere equivalent radius is used to compare two dimensions to three dimensions, a bias is introduced in the more ellipsoidal grains and grains with other nonspheroidal morphologies. This produces an overall bias in the lower tail. If the Saltykov correction were to be applied with a nonspherical geometry, this result could be improved. Furthermore, the lower tail is undersampled because caliper diameters below a limit are not included.

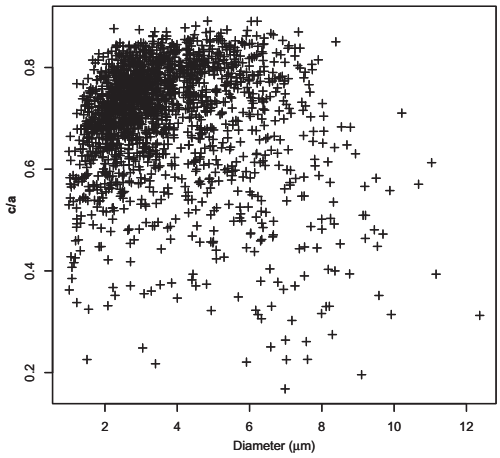

Figure 4. Plot of sphere equivalent diameter vs. aspect ratio (c/a) for the 3-D real grains of IN100. There are relatively more low aspect ratios in the small grain population, which is an underlying reason for the lower tail difference between 2-D and 3-D datasets.

Some of these intercepts may belong to a 3-D grain that is above the minimum size threshold. Corrections to the lower tail are left for future studies.

Although the entire range of grain sizes in three dimensions is not restored via the Saltykov method of spheres, the mean and upper tail significantly improve. This means that if the grain size, specifically the upper tail of the grain sizes, plays an important role in stress, strain, fatigue, etc. of Ni-based superalloys [19,20], a statistical volume element generated from 2-D data after applying Saltykov will more accurately predict a material response that depends on the upper tail of grain size.

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