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Three-Dimensional (3D) Analysis of Inclusions in Structural Alloys with Automated Serial Sectioning

Satya Ganti[1], Rachel Reed[1], William S. Davis[1] and Veeraraghavan Sundar[1]

[1] UES Inc., Robo-Met.3D, Dayton OH, USA.

Metallic and non-metallic inclusions (NMIs) have significant influence on the mechanical properties of materials [1]. These features in AISI 52100 bearings have been correlated to drastically reduce fatigue lives, particularly in wind turbine applications [2]. Characterization of inclusions for amount, size and shape distribution, chemical composition is of great interest for accurate determination of properties and aid in the quality control of materials manufacturing processes [3]. Serial sectioning is a practical and direct method of characterizing material feature size and shape distributions accurately in statistically relevant volumes of materials [4,5].

Two nickel- (Samples 1,2), two aluminum 6xxx series (Samples 3,4) and one steel (Sample 5) alloy sample were analyzed for this study. Samples were excised from actual parts, and were conventionally mounted in metallographic mounts for automated serial sectioning. Nickel and steel samples were analyzed for oxide/nitride/sulfide-based inclusions while AlFeSi intermetallics were studied in aluminum sample.

In this study, we used Robo-Met.3D, an automated system that metallographically generates optical microstructural data for 3D reconstruction in solid materials, with correlated SEM-EDS analyses to confirm the chemical composition of NMIs [4]. Binary images for 2D analysis were created by selecting pixel intensity threshold using Fiji/ImageJ, and were stacked and aligned using Fiji. Next, datasets with NMIs were reconstructed and visualized in 3D using Image-Pro Premier 3D software, version 9.3.

Two of the resultant 3D renderings are shown in Figure 1, visualizing size-classified inclusion distributions. The size distributions from the complete 3D experimental datasets captured with Robo-Met.3D are calculated and summarized in Table 2.

Critical parameters such as the difference between size distributions as well as morphologies in in inclusions were compared between samples. For example, Sample 3 had a higher percentage of fine (< 10 micron) particles, and a lower percentage of smaller aspect ratio particles (alpha AlFeSi, aspect ratio < 5) than Sample 4. This has an impact on mechanical properties.

For the steel sample, an inclusion with elongated morphology matching two classifications as defined by ASTM E45 (Type A-Sulfides and Type C-Silicates) was identified in a white etching region volume. The feature was traced for over 400 μ and had caused an in-service failure (Fig. 2).

The automated serial sectioning technique is being extended to study graphite clusters and carbide inclusions in cast iron and bearing steels.

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Sample / Alloy	Mag.	x-y Resolution	Per Layer	Analysis Volume
1 / Ni	100x	1.1 µ	5.9 µ	2000 x 2000 x 925 µ
2 / Ni	50x	2.1 µ	11.9 µ	1050 x 390 x 7140 µ
3 / Al	500x	0.2 µ	0.4 µ	200 x 150 x 60 µ
4 / Al	500x	0.2 µ	0.5 µ	200 x 150 x 65 µ
5 / Steel	500x	0.2 µ	5.6 µ	300 x 300 x 840 µ

Table 1: Sample and Imaging Details

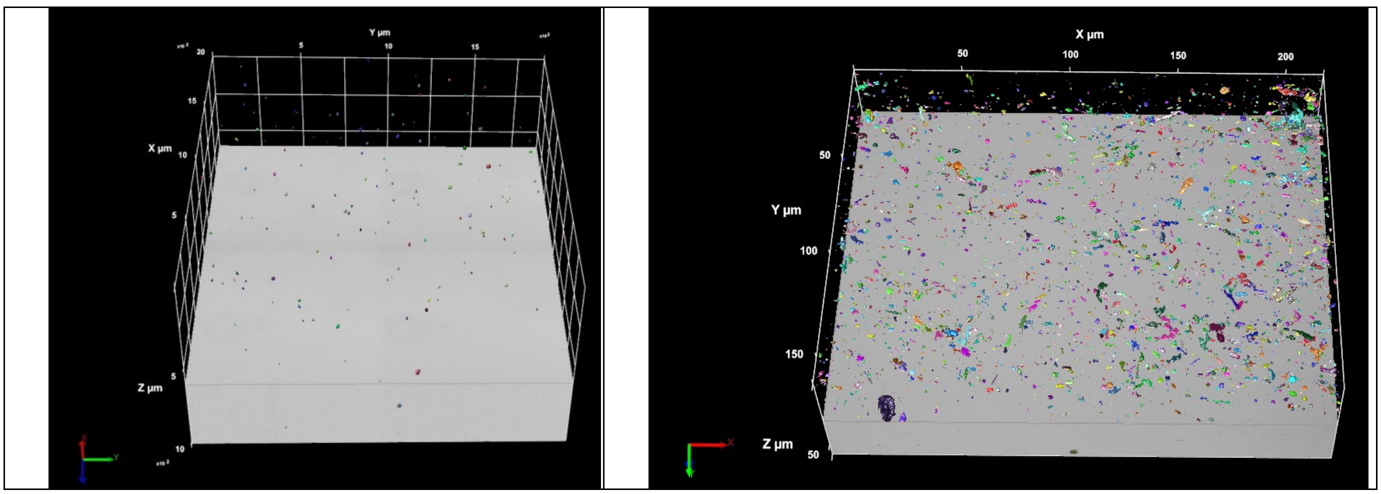

Figure 1. 3D NMI Distributions for Sample1 (left) and Sample3 (right)

Statistic	Sample 1	Sample 2	Sample 3	Sample 4
Minimum	5.4 µ	5.0 µ	0.3 µ	0.3 µ
Q1	6.9 µ	9.1 µ	0.7 µ	1.4 µ
Median	9.2 µ	12.3 µ	1.1 µ	2.5 µ
Q3	10.6 µ	19.6 µ	1.9 µ	4.5 µ
Maximum	21.5 µ	98.3 µ	28.7 µ	23.7 µ

Table 2. 3D NMI ESD Statistics for Selected Samples

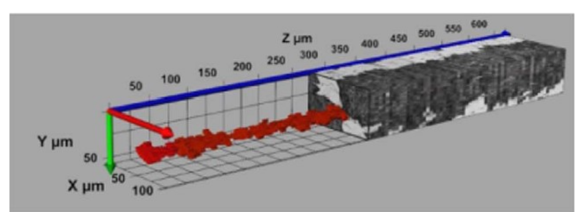

Figure 2. 3D rendering of the inclusion in segmented butterfly feature in M50 ball.

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