Advanced Digital Image Processing Technique for the samples processed in microgravity environment

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Introduction
The dihedral angle ($\Phi$) is formed by the lineal junction of solid-liquid interface and solid-solid grain boundary. During liquid phase sintering (LPS), wetting of the solid grain surfaces is usually controlled by dihedral angle. The 3D dihedral angle is the characteristic of the local interfacial energies of solid-liquid interface ($\gamma_{sl}$) and solid-solid interface ($\gamma_{ss}$) as shown by the Young’s equation $2\cos(\Phi/2)=\gamma_{sl}/\gamma_{ss}$. The objective of this project is to understand the effect of microgravity on various quantitative measurements like dihedral angle, volume fraction, mean-intercept length, and particle-size ratio for the samples LPS at different times (1, 45, 180 and 600 minutes).

Technical Approach
Samples of 50% W-35% Ni-15% Fe were liquid phase sintered at 1500°C in a microgravity environment of NASA’s space shuttle Columbus. The samples were prepared with different sintering times: 1, 45, 180 and 600 minutes. The samples were then sectioned, mounted, ground, and polished using SiC papers, diamond polishing (6 µm to 1 µm) and colloidal silica suspension (0.05 µm). Micrographs were obtained with magnification of 20X and 50X on unetched specimens to prevent error in section angle measurement. Digital image analysis software (KS-400) was used to convert grey scale image to the binary scale image (segmentation), which is then used to obtain various microstructural parameters like volume fraction, particle size distribution, dihedral angle, mean-intercept length, etc. The true mean dihedral angle in 3D microstructure was stereologically estimated from the measurements of section angles observed in 2D metallographic planes. A minimum of 500 angles were measured manually using a protractor as described by German [1]. The Interactive Data Language (IDL) software was used to generate the automated detection of triple points for automatic measurement of 2D dihedral angle. [2] IDL identifies the true triple junction along with some false triple points on a binary image. The most common false triple point detection seen was as a result of segmentation and due to the surface roughness as shown in Fig 1. The false triple point detected was manually separated from true dihedral angle using IDL. The procedure is relatively swift and enables a minimum of 1000 angles to be measured per hour.

![True Dihedral Angle](image1)

![False Dihedral Angle](image2)

Fig 1: Grey scale (left) binary (center) and processed image (right) of true and false positives dihedral angles

Results/Discussion
The automatic angle measurement shows the comparable mean between different sintering times. The automatic mean dihedral angle measurement for sintering times 1, 45, 180 and 600 min are 53°, 47°, 51° and 47° respectively. Figure 2 (A) compares the section angle distribution between 1 min and 600 min obtained from automatic measurements. The spread of section angle distribution in the 600 min specimen is lower compared to that of 1 min specimen, but it does have the comparable mean values of 50° and 51° between automatic and manual measurements. Figure 2 (B) compares the dihedral angle measurements taken manually with automatic measurement for the sintering time of 600 min. The mean grain size measured using the stereological techniques [3] are 6.2, 15.8, 21.9 and 31.3 µm respectively for 1, 45, 180 and 600 min samples. The average volume fraction of the four samples is 24%, which does not change with different sintering times.

![Dihedral Angle Distribution](image3)

**Figure 2**: Section angle distribution between 1 min and 600 min sample for automatic measurements (A) and manual vs. auto measurement comparison in 600 min sample (B)

Conclusion
The three-dimensional dihedral angle of W-grains has been estimated in the range of 50 ± 3 degrees using manual as well as automatic techniques. The mean dihedral angle does not vary with different sintering times (1 to 600 minutes). The mean intercept size of the W-grains on the other hand increases by a factor of 5 with the increase in sintering times.

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References