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# Estimation of three-dimensional mean dihedral angle in a W–Ni–Fe alloy liquid-phase sintered in microgravity

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The three-dimensional mean dihedral angle of impinging W grains is estimated in a 50 wt.% W–35 wt.% Ni–15 wt.% Fe alloy liquid-phase sintered in microgravity at 1500 °C for times ranging from 1 to 600 min using unbiased stereology and automated image analysis. The estimated mean dihedral angles are in the range $50 \pm 3°$. The mean dihedral angle does not vary significantly with the liquid-phase sintering time, although the mean intercept size of the W grains increases almost by a factor of 5.


*Keywords:* Liquid-phase sintering; Image analysis; Powder processing; Dihedral angle; Tungsten heavy alloys

The dihedral angle is the local angle between two microstructural surfaces where they meet to form a lineal edge element in the three-dimensional (3-D) microstructural space. A distribution of dihedral angles generally exists in microstructures. The mean value of the dihedral angle distribution is an important geometric attribute of the microstructure. The mean dihedral angle formed by solid–liquid interfaces at the necks (lineal junctions of two solid–liquid interfaces and a solid–solid grain boundary) is a geometric parameter that significantly affects microstructural evolution during liquid-phase sintering (LPS), which is a commonly used process for fabrication of near-net-shape components from powders [1]. During LPS, wetting of the solid grain surfaces by the liquid phase is primarily controlled by the dihedral angle at the necks. In general, smaller dihedral or wetting angles facilitate spreading of liquid over surfaces of solid grains and promote densification, but increase distortion in the liquid-phase sintered components [1]. The mean dihedral angle also affects the critical volume fraction for structural rigidity when the liquid phase is present [2] and the contiguity of the solid phase [3]. Further, the local equilibrium 3-D dihedral angle $\chi$ at a neck in an LPS microstructure is related to the ratio of the local interfacial energies of solid–liquid interfaces $\gamma_{S\text{–}L}$ and the solid–solid grain boundary energy $\gamma_{S\text{–}S}$ through Young's equation given below:

$$2\cos(\chi/2) = \gamma_{S\text{–}S}/\gamma_{S\text{–}L} \quad (1)$$

Thus, if the equilibrium dihedral angle $\chi$ is experimentally measured, the interfacial energy ratio $\gamma_{S\text{–}S}/\gamma_{S\text{–}L}$ can be calculated. Consequently, estimation of the mean dihedral angle is of interest for measurements of the average interfacial energy ratio as well as to understand and model microstructural evolution during LPS.

Direct estimation of true 3-D dihedral angles is difficult except in the special cases where the particles/grains can be selectively dissolved and the particle shape can be modeled as generated by two intersecting spheres of the same size [4,5], which is usually not true in the LPS microstructures. An apparent dihedral angle observed in a two-dimensional (2-D) metallographic section depends on both the corresponding true 3-D dihedral angle and the angular orientation of the sectioning plane, and therefore it is not necessarily equal to the corresponding true 3-D dihedral angle. Consequently, a distribution of apparent 2-D section dihedral angles is observed in random 2-D metallographic sections even when there is only one true 3-D dihedral angle. Nevertheless, the true mean dihedral angle in the 3-D

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microstructure can be stereologically estimated in an assumption-free and unbiased manner from the measurements of section angles (apparent dihedral angles) observed in uniform random 2-D metallographic planes. The stereological problem of estimation of true mean 3-D dihedral angle from the measurements of 2-D section angles was first analyzed by Harker and Parker [6], who derived the relationship between the apparent dihedral angle (section angle) resulting from an intersection of a metallographic plane of any given orientation and a dihedral angle of any given measure in the 3-D microstructure. Subsequently, DeHoff [7], Reeds and Butler [8,9] and Miles [10] showed that the mean value of the 2-D section angle distribution is always an unbiased estimator of the mean value of the 3-D dihedral angle distribution, and the relationship is applicable to any arbitrary microstructure provided that the sampling is isotropic, uniform and random (IUR). It is essential to emphasize that it is the mean value of the section angle distribution that is an unbiased estimator of the true mean 3-D dihedral angle: the mode and the median of the section angle distribution are not necessarily unbiased estimators of the true mean 3-D dihedral angle. The distinction is important because section angle distributions are often skewed, and therefore the mean, the mode and the median of the section angle distribution are not necessarily equal.

Stereological estimation of the mean 3-D dihedral angle from 2-D section angle measurements requires unbiased and representative statistical sampling, and therefore a sufficiently large statistical sample of section angles (~1000 angles) is needed for reliable estimation (small sampling error) of the mean value of 3-D dihedral angle. Experimental measurements of mean dihedral angle have been reported in numerous LPS alloys [11–15]. However, in the majority of these studies, the measurements were performed manually, and consequently, a relatively small number of section angle measurements were performed (~100). The dihedral angle measurements in the LPS microstructures have led to the conclusions that the mean 3-D dihedral angle systematically varies with the grain size or processing time [11,12,14,15] and volume fraction of the solid phase (grains). The reasons for such variations in the mean dihedral angle are not clear. Further, earlier estimations of the mean dihedral angles in the LPS microstructures are in the microstructures containing solid volume fractions higher than 40%. There are no data on the mean dihedral angles in the LPS microstructures containing low volume-percentage of the solid phase. Such data are useful for understanding the dependence of the mean dihedral angle on the volume-percentage of the solid phase in the LPS alloys.

This contribution concerns estimation of the 3-D mean dihedral angle of impinging W grains in 50 wt.% W–35 wt.% Ni–15 wt.% Fe alloy liquid-phase sintered at 1500 °C in the microgravity environment of NASA's space shuttle *Columbia*. In this alloy, the volume-percentage of the W grains is low (24%). Microgravity material processing of the alloy eliminates solid–liquid separation due to gravity that can lead to microstructural heterogeneity in the spatial distribution of the W grains and other artifacts that can bias the microstructure. In the present work, in each specimen, measurements of more than 1000 section angles have been performed using automatic image analysis for reliable estimation of the true 3-D mean dihedral angle. The data reveal that there are no statistically significant variations in the mean dihedral angle of the W grains with the LPS time from 1 to 600 min, although the mean intercept grain size of the W grains increases almost by a factor of 5 in this time interval. Therefore, it is likely that some of the variations in the estimated 3-D dihedral angles with LPS time (or with grain size) reported in earlier studies were due to the large sampling errors and the measurement errors associated with manual measurements of small number of section angles (~100).

The specimens were prepared from powders of tungsten (Osram Sylvania M37, median size 12.1 μm), nickel (Novamet INCO 123, median size 11.2 μm) and carbonyl iron (ISP R1470, median size 6.0 μm). The samples of 50W–35Ni–15Fe (wt.%) composition were cold isostatically pressed from dry-blended elemental powders at 200 MPa into cylinders 12.7 mm in diameter and ~10 cm long. Pressed compacts were then vacuum pre-sintered at 1400 °C for 1 h. The pre-sintered compacts were sealed into cylindrical alumina crucibles under a vacuum of $10^{-4}$ Torr or better and flown aboard shuttle *Columbia* for microgravity sintering. In the microgravity environment, the samples were heated at rate of 10 °C $min^{-1}$ to 800 °C; held at 800 °C for 60 min; ramped at 10 °C $min^{-1}$ to 1500 °C; and held at 1500 °C for different times ranging from 1 to 600 min. The specimens were then cooled at the rate 3 °C $min^{-1}$ to 1420 °C and then furnace cooled to ambient temperature. The sintering atmosphere was a static vacuum, nominally $10^{-4}$ Torr pressure. It should be noted that the thermal profile used in sintering resulted in a total time over solidus (1430 °C) of isothermal hold time plus 30 min. The details of these microgravity experiments are reported elsewhere [16]. The dihedral angle measurements have been performed on the specimens liquid-phase sintered at 1500 °C for 1–600 min in microgravity.

The specimens were sectioned along the cylinder axis approximately at the center and then mounted in metallographic mounts. The grinding and polishing steps involved grinding on SiC papers (240, 400 and 800 grit) followed by diamond polishing (6–1 μm) and finally polishing with colloidal silica suspension (0.05 μm size). The section angle measurements were performed on unetched specimens because etching may alter the local morphology at the necks that can lead to bias (systematic error) in the section angle measurements. Figure 1(a) shows the unetched microstructure of the specimen liquid-phase sintered at 1500 °C for 1 min, and Figure 1(b) depicts the microstructure of the specimen held for 600 min at 1500 °C. As expected, the microstructural length scale and the mean grain size of W grains in the 600 min specimen are substantially higher than that in the 1 min specimen due to grain coarsening. Quantitative microstructural analysis was performed on high-resolution (~1 μm) large-area (2–10 $mm^2$) microstructural montages generated by stitching together numerous contiguous microstructural fields using the procedure described in detail elsewhere [17–19].