Chapter 2
Characterization of Microstructure in Semisolid Slurries

It has been understood for some time that the flow behavior of semisolid metal slurries, and the properties of parts shaped from the slurries, depend both on the fraction solid and on the size, distribution, and morphology of the solid particles within the liquid matrix. The fraction solid $f_s$ is essentially determined by the temperature for a given alloy; the other features, however, are a function of the history of preparation and it is known that slurry flow is enhanced by achieving fine spherical particles.

2.1 Fraction Solid

Fraction solid ($f_s$) can be determined from phase diagrams for simple alloy systems if equilibrium may be assumed, or from direct measurement on rapidly quenched specimens, using metallographic techniques on sectioned surfaces, such as line intercept: $f_s = L_a$, where $L_a$ is the total intercept length in the a particle phase per unit length of test line. Likewise, the average particle size (assuming spheres of constant diameter $d$) may be obtained by the measurement: $d = L_a/N_a$, where $N_a$ is the number of grains per unit area of sectioned surface. It should be noted that the fraction solid obtained from phase diagrams is strictly a weight fraction and only coincides with the volume fraction obtained from metallographic measurements above when the densities of liquid and solid are considered equal and shrinkage is therefore ignored. Other techniques have been used to obtain these quantities, but are indirect, requiring calibration, and are probably less reliable. However, see in situ X-ray microtomography in Sect. 3.4, where the measurement is carried out in the semisolid condition to avoid quenching artifacts.

2.2 The Shape Factor

Spheroidized particles within a semisolid alloy slurry are an essential prerequisite structure for effective thixoforming, and it is necessary therefore to develop a quantitative description of this property. One commonly used descriptor, which may be obtained from two-dimensional sections through a complex morphological structure, is to measure the perimeter length $P$ of an object particle present from a
section, and its sectioned surface area $A$. The dimensionless shape (or form) factor is then defined as: $F_o = P^2 / 4 \pi A$ (note that some authors use the reciprocal of this factor); in the case of a section, which appears as a circle, $F_o = (2 \pi r)^2 / 4 \pi \cdot r^2 = 1$, and convoluted perimeters would clearly produce values in excess of unity. However, it is clear from the diagram in Fig. 2.1 that circular sections may not only arise from spheres, but also from much more complex morphologies in three dimensions, such as sections through cylindrical dendrite arms, and such complex morphologies are not reflected in this simple shape factor.

To overcome this drawback, Loué and Suery [13] have introduced a different dimensionless shape factor defined as: $F_g = 1 / (6 \pi f_s) \left( S_v^2 / N_a \right)$, where $S_v$ is the surface area per unit volume of the particulate phase and $N_a$ the number of grains per unit area of sectioned surface. Both of these values are in principle able to be measured by direct metallographic techniques from two-dimensional sections. For the situation of a monodisperse array of spheres having a uniform diameter $d$, we have:

$$S_v = \pi d^2 N_v = \pi d^2 \cdot 6 f_s / \pi d^3 = 6 f_s / d$$

and

$$N_a = N_v d = (6 f_s / \pi d^3) d = 6 f_s / \pi d^2$$

so that

$$F_g = \frac{1}{6 \pi f_s} \left( \frac{6 f_s}{d} \right)^2 \left( \frac{\pi d^2}{6 f_s} \right) = 1.$$
Some insight into the physical meaning of this shape factor may be gained from the following simple consideration. Imagine a simple square array of cylindrical dendrite side arms, all belonging to the same grain. If each arm has a diameter, \( d \), and the arms are separated by a distance \( \lambda \) (see Fig. 2.1), then \( f_s = \pi d^2/\lambda^2 \), the interface area per unit volume \( S_v = \pi d/\lambda^2 \) (that is inversely proportional to \( \lambda \) for a given \( f_s \)) and the number of grains per sectioned surface area \( N_a = 1/(n\lambda)^2 \), where \( n\lambda \) is approximately the grain size.

Hence \( F_g = \frac{1}{6\pi f_s N_a} = \frac{1}{6\pi f_s} \left( \frac{\pi d}{\lambda^2} \right)^2 (n\lambda)^2 = \frac{2}{3} n^2 \), so that this shape factor is simply related to the number of side arms possessed by a dendrite. For \( n = 10 \) side arms, \( F_g = 67 \), which is similar to the value observed in a conventionally DC cast billet, see Fig. 3.1a. An anomaly exists for a single independent cylindrical rod, where \( n = 1 \), giving \( F_g = 0.67 \), smaller than the value for a sphere; however, a rod can hardly describe a dendritic array and appears the consequence of an oversimplified model not intended to predict the complete morphological change of a dendrite with time.

Any dispersed system will tend to minimize its surface free energy and therefore, its surface area with time. This requires that in complex convoluted morphologies with high surface energy per unit volume \( (S_v) \), the particles will first spheroidize (i.e., \( F_g \to 1 \)), followed by the process in which the smaller particles or grains dissolve to feed the larger ones (akin to Ostwald ripening). The first stage will proceed more rapidly because of the shorter diffusion distances involved, and the objective of the heating stage for thixoforming is to achieve this first stage of spheroidization without entering the second stage of grain growth.

2.3 Contiguity and Continuity

Gurland [14] has introduced the concept of contiguity ratio defined as the average fraction of the surface area of a particle shared with neighboring particles of the same phase:

\[
C = \frac{2S_{aa}}{2S_{aa} + S_{ab}},
\]

where \( S_{aa} \) is the area of interface between \( \alpha \) particles, \( S_{ab} \) between \( \alpha \) particles and the \( \beta \) matrix and the factor 2 appears because for contiguous particles the interface are counted only once. For completely isolated particles, \( C = 0 \), whereas for grains in total contact (i.e., \( f_s = 1 \)), \( C = 1 \). This parameter is not a function of the shape or size of the particle; it is concerned only with contacts between grains and their immediate neighbors and may be regarded as a measure of short-range continuity as experienced in particle clustering. It is expected to depend on both processing and subsequent heat treatment.
20 Characterization of Microstructure in Semisolid Slurries

---

**Table 1. Experimental Measurements on Silver-Bakelite Aggregates**

<table>
<thead>
<tr>
<th>Composition, vol pct Ag</th>
<th>Mean Number of Contacts per Silver Particle</th>
<th>Electrical Resistivity, ohm-cm</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.40</td>
<td>$3.5 \times 10^9$</td>
</tr>
<tr>
<td>20</td>
<td>1.00</td>
<td>$2.5 \times 10^9$</td>
</tr>
<tr>
<td>30</td>
<td>1.25</td>
<td>$5.0 \times 10^9$</td>
</tr>
<tr>
<td>36</td>
<td>1.33</td>
<td>$1.2 \times 10^9$</td>
</tr>
<tr>
<td>37</td>
<td>1.52</td>
<td>10</td>
</tr>
<tr>
<td>38</td>
<td>1.45</td>
<td>$9.2 \times 10^8$</td>
</tr>
<tr>
<td>40</td>
<td>2.47</td>
<td>$3.1 \times 10^8$</td>
</tr>
<tr>
<td>50</td>
<td>2.74</td>
<td>$2.9 \times 10^8$</td>
</tr>
</tbody>
</table>

---

**Fig. 2.2**

![Graph showing log of resistivity vs. volume percent, silver](image-url)
Gurland [15] has used a different measure to describe the degree of long-range continuity of a dispersed phase within a matrix, defined as the probability of infinitely long chains of connected particles being formed, which are orientated randomly and cross-linked in 3D, to provide a rigid skeletal framework. This is based on the average number of contacts \( \bar{m} \) that a particle makes with neighboring particles of the same phase. Experimental measurements on compacts of silver spherical particles embedded in bakelite show that the resistivity falls off dramatically as the volume fraction of silver increases from 0.36 to 0.38 (Fig. 2.2). This is interpreted to indicate that long chain structures develop rapidly over this compositional range. The average number of contacts of a silver particle were measured by quantitative microscopy on specimens in this region, and it was established that long range continuity occurred when \( \bar{m}^* > 1.5 \). This critical value for \( \bar{m} \) is supported by two simple models investigated, a branching chain model and a percolation model, both of which also demonstrate the dramatic transition to long range chain structures with composition. In the light of the above, it would appear that for the conditions under which semisolid forming is carried out at \( f_s = 0.5-0.6 \), long-range skeletal structures may be expected to be created, thus contributing to the observed stiffness in these materials.

In a later paper, Lee and Gurland [16] introduced the concept of “continuous volume” to explain the hardness of cemented carbides. They showed that the yield strength \( \sigma \) of a composite material containing a skeletal structure of particles in a continuous matrix could be given by:

\[
\sigma = \sigma_p V_c + \sigma_m (1 - V_c),
\]

where \( \sigma_p \) and \( \sigma_m \) are the yield stresses for the particulate and matrix phases respectively, and \( V_c \) is the fraction of continuous volume of the particulate phase bearing the load shown to be equal to \( CV \), where \( C \) is the contiguity ratio and \( V \) the volume fraction of particulate. This expression appears to describe the hardness of composites containing tungsten carbide particles in a cobalt matrix very well. If we use it for semi-solid slurries, \( \sigma_m = 0 \), since the liquid matrix will bear no load, and writing \( f_s \) for \( V \), we have \( \sigma = \sigma_p C_f \) where \( C_f \) is the continuous volume fraction of the solid skeleton. Inserting a value of \( \sigma_p \approx 2 \text{MPa} \) (obtained from extrapolating high temperature data for pure aluminum [17] to 570°C), and using experimental measurements of \( C_f = 0.15 \) [18], we obtain \( \sigma \approx 0.3 \text{ MPa} \), which is more than sufficient to prevent sagging of a slug of 10 cm height under gravitational force (~3 kPa).
Semi-solid Processing of Alloys
Kirkwood, D.H.; Suéry, M.; Kapranos, D.P.; Atkinson, H.V.; Young, K.P.
2010, XII, 172 p. 103 illus., 19 illus. in color., Hardcover
ISBN: 978-3-642-00705-7