Single fluid atomization through the application of impulses to a melt

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Abstract

Impulse Atomization (IA) is a single fluid atomization technique that is capable of producing droplets of controlled size having a relatively tight distribution and a predictable cooling rate. The process has been successfully employed to produce a wide range of metal droplets including Pb–Sn alloys, aluminum alloys, copper alloys, low carbon steel and tool steel. Atomization characteristics determined from load cell measurements, video imaging and particle size analysis are discussed as a function of process characteristics. It is shown that atomization occurs by Raleigh instability and that only primary atomization of the stream is in effect. The rate of cooling of a moving molten droplet has been modeled and experimentally validated using this atomization technique. The droplet Nusselt number ranges nearly from 2 to 10, indicating that thermal conduction from the droplet to the gas is an important mechanism by which the droplet loses heat. Comparison of droplet microstructure of IA and gas atomized powders reveals that for the same size powder of the identical alloy, IA generates a finer microstructure or solidifies with a higher cooling rate. This is attributed to two-way thermal coupling (between gas and melt spray) in gas atomization being greater than in IA. These atomization and heat flow characteristics clearly demonstrate a number of unique features of this technique as well as its flexibility to meet different processing requirements for production and research. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Atomization; Rapid solidification; Heat transfer

1. Introduction

In recent years, atomization of melts (high temperature fluids such as metals, alloys and slags) has seen significant innovative new breakthroughs, developments and capabilities. These are primarily with the single-fluid atomization approach where a melt stream is rendered unstable by Raleigh instability in a static gas atmosphere. Under the right atomization conditions, a mono-size or a controlled and narrow size distribution of droplets is generated. The droplets either are allowed to fall through a stagnant gas atmosphere and solidify or are deposited onto a substrate.

Recently, a number of groups worldwide have reported new breakthroughs in the single fluid atomization of melts. The first is based on the drop-on-demand technique [1]. This is essentially the same atomization technique used in ink jet printers. In this method, a fluid wets a micron-sized orifice by capillary action. Through the application of pressure on a diaphragm, the fluid is ejected from the orifice forming a single droplet. The repeated application of this pressure at high frequency and low amplitude allows the generation of a sequence of droplets. This method of atomization has to date been limited to low melting point melts (Pb and Sn) and to millimeter sized granules principally due to difficulties in developing construction materials for high temperature operation.

Two groups [2,3] base their approach on vibrating a continuous column of melt into micron sized monodroplets. Used to date only for low temperature alloys (up to Al), this method generates a stream by applying gas pressure over the melt. The melt stream is vibrated using various approaches to allow breakup of the jet into mono-sized droplets that are as close as one particle diameter apart in flight. To avoid droplet collision, since the droplets are decelerating to their terminal velocity, special techniques are used to disperse the
which provide a very reliable approach to developing cell spacing versus cooling rate relationships. Finally, the microstructure from IA is compared with those generated from gas atomization.

2. Atomization characteristics

Impulse Atomization (IA) [5,6] is a single fluid atomization technique in which tight control over size and shape and cooling conditions can be achieved. Schematics of the process and of the experimental apparatus are shown in Figs. 1 and 2. Included in these figures are the impulse generator, the mechanical oscillator (driver), a metal tundish system and a nozzle plate complete with orifices. Depending on the type of product one wishes to generate, one of three modes of operation can be selected with the IA technique. These modes are atomization, impulse and impulse/gravity. By varying the plunger acceleration, orifice size, orifice shape, melt temperature and gas atmosphere, the size, distribution, shape and microstructure can be manipulated to the required specifications. The quiescent gas atmosphere into which the melts have been atomized include helium, nitrogen, argon and air. To date powders and granules have been produced in the 0.5 × 4 m experimental tower with a tailored mass median particle size (D_{50}) and a controlled log-normal standard deviation (S.D.) (D_{84}/D_{50}) of 1.1 ≤ σ ≤ 1.6 using two of the three modes of operation (impulse and gravity/impulse). Atomization in the impulse mode has been carried out with as many as 97 orifices in a 2.5 cm
The size of granules or powder produced is governed by several variables including the diameter of the atomizing orifices. For granule production, the stream velocity emanating from the orifice is governed by the melt head and the plunger action. The plunger also provides the required disturbance to generate controlled break-up of the stream. When producing granules, of course, special cooling techniques must be used to cool the granules in a reasonable distance. Fig. 3 displays the mass mean ($D_{50}$) particle size as a function of orifice diameter for aluminum, copper and iron alloys produced using the impulse mode. The solid line shown in the graph represents a linear regression analysis of the data. From the slope of this line, the ratio between particle size and orifice diameter is about 1.28. The range in $D_{50}$ observed at a constant orifice diameter is attributed in part to the current batch-mode operation of the atomizer, ranging typically from 0.5 to 2 kg. In addition, the log-normal S.D. of particle size ($D_{84}/D_{50}$) for the same alloys and orifices sizes is shown in Fig. 4. For the alloys atomized, the S.D. lies primarily between 1.3 and 1.5. The results at about 1.9, 1.7 and 1.65 are considered outliers and occurred during early atomization trials with the iron, copper and aluminum alloys, respectively.

Unlike other single fluid atomization techniques [2,3], IA generates in the impulse mode of operation a disturbance on a segment of fluid which break up into several drops. Using the measured mass flow data in IA coupled with a sieve analysis, the number of particles produced for each atomization pulse ranges between 12 and 13 particles. It should be noted that this number includes particles in the finer range of the distribution, which in terms of mass make up a very small proportion of the overall total particles. In comparison, the minimum theoretical wavelength [7,8] (according to linear capillary instability) of the disturbance required for break up of a liquid stream is $\lambda = \pi d$, where $d$ is the diameter of the ligament. For the 500 μm orifice atomization orifice plate. Most recently, steel powders have been successfully atomized with IAP with the $D_{50}$ particle size ranging from 354 to 1508 μm. Using the impulse/gravity mode aluminum monodisperse granules have been produced having sizes from 2 to 10 mm. Depending on melt chemistry and gas atmosphere powder shape can be controlled including the generation of spherical particles. In Table 1, a summary of the type of materials and range of powder and granule sizes produced to date with IA is presented. Standard refractory materials are used for the tundish, plunger and orifice plate.

### Table 1

<table>
<thead>
<tr>
<th>Metal or Alloy</th>
<th>$D_{50}$ size range (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>310, and 2, 4, 6, 8, 10 mm</td>
</tr>
<tr>
<td>Al-5% Cu</td>
<td>400</td>
</tr>
<tr>
<td>Al-10% Cu</td>
<td>364–517</td>
</tr>
<tr>
<td>Al-17% Cu</td>
<td>381–515</td>
</tr>
<tr>
<td>Al-24% Cu</td>
<td>377–521</td>
</tr>
<tr>
<td>Al 6061</td>
<td>250–850</td>
</tr>
<tr>
<td>Al-10% Sr</td>
<td>1000</td>
</tr>
<tr>
<td>Al-24% Sr</td>
<td>1000</td>
</tr>
<tr>
<td>Al 357</td>
<td>549–692</td>
</tr>
<tr>
<td>Copper</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>200–1400</td>
</tr>
<tr>
<td>Bronze (90/10)</td>
<td>400–800</td>
</tr>
<tr>
<td>Pb-10% Sn</td>
<td>200–1000</td>
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<tr>
<td>Pb-12% Sn</td>
<td>130</td>
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<tr>
<td>Lead</td>
<td></td>
</tr>
<tr>
<td>Mg</td>
<td>850–1000, and 2 to 4 mm</td>
</tr>
<tr>
<td>Mg-9Al-1Zr</td>
<td>850</td>
</tr>
<tr>
<td>Steel</td>
<td></td>
</tr>
<tr>
<td>Fe-W alloy</td>
<td>1425</td>
</tr>
<tr>
<td>1040</td>
<td>1508</td>
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<tr>
<td>4140</td>
<td>354</td>
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<tr>
<td>4140</td>
<td>881</td>
</tr>
<tr>
<td>H-13</td>
<td>538</td>
</tr>
<tr>
<td>Neodymium</td>
<td></td>
</tr>
<tr>
<td>NdFeB</td>
<td>100</td>
</tr>
<tr>
<td>Zinc</td>
<td>Zn-500 ppm Pb</td>
</tr>
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</table>

Fig. 3. Mass mean ($D_{50}$) particle size as a function of atomizing orifice diameter.

Fig. 4. Log-normal standard deviation as a function of orifice size.
izing aluminum, the minimum $\lambda$ value is equal to 1.57 mm. If each wavelength is assumed to form a single droplet then, considering that the length of the ligament emitted from the orifice is on the order of 4–5 mm (Table 2) one would expect the formation of three to four droplets per discrete stream [7]. The difference between the actual number of particles (12–13) formed per pulse and the value predicted by ligament break-up theory (3–4) is attributed to a large number of finer particles (which represents a very small mass fraction). These seem to form during primary atomization. The challenge of our current research is to provide even greater control on the distribution while increasing throughput [9].

In addition studying the breakup mechanism in IA, the mass flow data was used to record the productivity of IA. Measured mass fluxes for aluminum range at present between 2000 and 3000 kg m$^{-2}$ s$^{-1}$. This mass flux, which is based on the orifice area, varies with a number of process variables and is being optimized. However, this compares favorably with the mass flux achievable in gas atomization. The initial droplet velocity was also estimated from the mass flow rate measurements and was measured using freeze-video imaging coupled with a mathematical model of the droplet trajectory. Both approaches yielded a similar initial droplet velocity of about 0.5 m s$^{-1}$ for a wide range of droplet sizes [9].

### 3. Solidification characteristics

IA powders are rapidly solidified. In characterizing the microstructure of a 700 µm particle of IA:Al–24 wt.% Cu atomized in helium, Dr Ke Han, Los Alamos, clearly identified using an FESEM regions of nucleation, recalescence and dendritic growth. Subsequent studies on phosphorous bronze using SEM also displayed similar features as shown in Fig. 5.

### Table 2
Mass flow rate data [7]

<table>
<thead>
<tr>
<th>Fraction acc.</th>
<th>Mass flow per orifice (g s$^{-1}$)</th>
<th>Ligament length (mm)</th>
<th>Exit velocity (m s$^{-1}$)</th>
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<tr>
<td>980730</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>0.375</td>
<td>0.252</td>
<td>5.35</td>
<td>1.07</td>
</tr>
<tr>
<td>0.500</td>
<td>0.200</td>
<td>4.24</td>
<td>0.85</td>
</tr>
<tr>
<td>0.625</td>
<td>0.218</td>
<td>4.64</td>
<td>0.91</td>
</tr>
<tr>
<td>0.750</td>
<td>0.225</td>
<td>4.80</td>
<td>0.95</td>
</tr>
<tr>
<td>0.875</td>
<td>0.218</td>
<td>4.69</td>
<td>0.93</td>
</tr>
<tr>
<td>980804</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.375</td>
<td>0.039</td>
<td>1.42</td>
<td>0.284</td>
</tr>
<tr>
<td>0.500</td>
<td>0.056</td>
<td>2.03</td>
<td>0.407</td>
</tr>
<tr>
<td>0.625</td>
<td>0.069</td>
<td>2.53</td>
<td>0.506</td>
</tr>
<tr>
<td>0.750</td>
<td>0.109</td>
<td>3.97</td>
<td>0.795</td>
</tr>
</tbody>
</table>

Fig. 5. IA phosphorous bronze powder atomized in nitrogen at 955 °C (run #971031).

Fig. 6. Volume Fraction of CuAl$_2$ in helium and nitrogen atomized granules as a function of copper content [10].
In a study on the effect of the homogeneous distribution of CuAl$_2$ phase in an Al matrix, the volume fraction of the eutectic phase in a binary Al–Cu alloy was measured by image analysis on IA atomized powders [10]. The results are shown in Fig. 6 along with the equilibrium volume percent of CuAl$_2$ given by the Al–Cu equilibrium Phase Diagram at the eutectic temperature of 548 °C. It is evident that the experimental results of the 5 and 10 wt.% Cu are above the equilibrium line while those at 17 and 24 wt.% Cu are below. Note that in the metastable phase diagram proposed by Gill and Kurz [11], both the 5 and 10 wt.% Cu are in the single phase region. Thus, the higher CuAl$_2$ content is consistent with microsegregation of Cu during solidification. At the higher Cu contents, the lower than equilibrium values of CuAl$_2$ are consistent with solute trapping mechanisms in rapid solidification [12]. From all these observations it is evident that IA is yielding a rapidly solidified microstructure.

4. Thermal cooling rate of droplets

For metal powder production or spray deposition, an important process parameter is the heat transfer coefficient or cooling conditions to which a moving molten droplet is subjected. It is well known that the fineness of a structure (cell or dendrite arm spacing) is inversely proportional to the rate of heat extraction during solidification [13]. This relation takes the form:

$$\lambda = Be^{-n}$$

(1)

where $\lambda$ is the cell or dendrite arm spacing, $e$ is the cooling rate during solidification and $B$ and $n$ are constants determined from a best fit analysis. Our ability to control $\lambda$ is directly related to the cooling rate during solidification of the droplet. The cooling rate of atomized droplets in flight also affect the spray deposition process by governing solidification distances of the atomized droplets relative to deposition distance. In view of its importance, an accurate understanding of the heat transfer coefficient prevalent at the surface of a moving droplet is essential.

Given the complexity of atomization sprays, heat transfer analysis has been generally confined to the study of a single droplet moving with respect to a gas. The sophistication of heat transfer analysis ranges from complex numerical simulations [14] in which the fluid flow and energy equations are explicitly solved for the moving droplet to a more mechanistic approach based on semi-empirical equations. In this latter grouping, equations describing heat transfer conditions (Nusselt number) as a function of a droplet’s relative velocity and the thermophysical properties of the cooling medium have been developed (e.g. Ranz–Marshall [15] and Whitaker [16]).

Owing to their ease of use, these relations have been used by a number of researchers to study particle cooling during atomization [17–21]. Experimental validation of the published models is seldom executed due to the difficulty in undertaking the required controlled experiments. Stone and Tsakiropoulos [22] attempted to correlate cell spacing measurements measured for a gas atomized Al–4.5% Cu alloy with droplet cooling rate determined by calculating droplet heat transfer. They reported an order of magnitude difference between measured and predicted cooling rates. Both correlations given by Ranz–Marshall and Whitaker were derived by regression fitting several of the equations parameters to experimental data [15,16]. Though directly applicable to the test conditions from which they were correlated, the appropriateness of these equations for high temperature atomization is not well understood. In particular, the influence of the gas thermophysical properties used in calculating the Re and Pr number and of the conductivity of the gas in the Nusselt number on the effective heat transfer calculation needs to be understood.

The IA technique offered a unique opportunity to validate different models of gas-droplet solidification. Using a quench test, the droplets that were solidified after a specific distance of trajectory was determined for AZ91D$^1$ and AA6061$^2$. Droplets with a mixed coarse and fine dendritic structure were indicative of droplets that did not solidify entirely in the gas phase. Those with a solely a coarse structure clearly were fully solid in the gas phase before entering a quench bath. The experimental procedure is described in detail elsewhere [9,23].

Using the heat flow model [24] and the heat transfer coefficient calculated using either the Whitaker or Ranz–Marshall equations, the cooling rate as a function of particle diameter was determined for both atomizing in argon and nitrogen. Except where noted, the Re and Pr numbers used in calculating the Nusselt number were all determined using the gas thermophysical properties evaluated at the ambient temperature (300 °K). The thermophysical properties for nitrogen and argon [25–28] and for the AZ91D alloy [27,29] were used in the model as a function of temperature. It has been clearly shown that the Whitaker correlation shows good agreement with the experimental results when the thermal conductivity of the gas is evaluated at the droplet temperature. This is consistent with the fundamental definition of the Nusselt number [30], which is the ratio of the total heat transfer between

---

1 Composition of AZ91D (wt.%): 7.76Al; 1.16Zn; 0.307Mn; 0.157Cu; 0.076Si; 0.045Fe; 0.00042Ni.

2 Composition AA6061 (wt.%): 0.25Cu; 0.96Mg; 0.64Si; 0.19Cr; 0.05Zn; 0.46Fe.
It is clear from the results in Fig. 7 that conduction between droplet and gas accounts for at least 25% of the heat transfer mechanism. It should be noted that although the Nu is small, the calculated heat transfer coefficient for a given droplet size is very large. Hence, the heat transfer coefficient between droplet and gas is a strong function of the size and the temperature of the droplet and the gas type into which the droplet is falling and losing heat. It is equally evident that in IA the role of the gas is primarily to remove heat from the droplets. This analysis clearly shows that using IA very controlled heat transfer is obtained between droplet and gas. Given the small droplet dimensions, which eliminate the presence of macrosegregation, IA offers a novel and reliable technique to determine cell spacing versus cooling rate correlation.

The calculated droplet cooling rates in IA using the thermal model that was experimentally verified and described above were plotted against the measured cell spacing for an Al–4.5 wt.% Cu. These results are shown in Fig. 8(a) and compared with the regressed equation proposed by Young and Kirkwood [32] who used the Bridgeman technique to analyze the same alloy [33]. The ingot they used was relatively small in cross section minimizing the occurrence of macrosegregation. This is unlike earlier workers whose results are shown in Fig. 8(b) whose samples were very large (> 2.5 cm in diameter) or who estimated the heat transfer coefficient to the cooling mold. Clearly, IA is a reliable technique for determining cell spacing versus cooling rate relationship [33–36]. Due to the controlled heat transfer between each droplet and gas, the size of the droplet and the reproducible atomizing conditions, IA shows promise as a unique technique to explore the fundamentals of microsegregation and to incorporate diffusive mass transfer in the determination of solidification cooling rate. It also provides an opportunity to better understand other atomization techniques. Current results indicate that IA provides a finer structure than the equivalent powder sized particle in gas atomization as will be discussed below.

5. Comparison of atomization processes

Since IA provides a predictable and controlled heat transfer rate between droplet and gas, a comparison is carried between powder microstructures obtained with IA and those generated by other atomization processes. The first such comparison is shown in Fig. 9 for an Al–Fe–Ni alloy [37]. Three atomization processes are compared: gas atomization (Alcoa), centrifugal atomization and IA. While IA powders are coarser in size than those of the other two processes, an interesting trend emerges. It is well known that the relationship between dendrite arm spacing and particle size is linear.
different behavior, IA powder microstructures appeared to be very fine given the large size of the powder particles.

A comparison was carried out between horizontal gas atomization and IA using spherical bronze powders (see Fig. 10). In horizontal gas atomization, the melt temperature is 1150°C and atomization is carried out in air. Comparing the microstructures from gas atomization to those from IA with a melt temperature of 1237°C, the IA powders are clearly considerably finer. Also note the significantly smaller exponent in the two curve fitted equations (0.521 for gas atomization versus 0.426 for IA). This smaller slope is similar to what was observed with the IA Al–Fe–Ni alloy earlier in Fig. 9.

In examining the gas-atomized powders, it was noted that most particles contained two types of microstructures similar to that shown in Fig. 5. Hence, the results shown in Fig. 10 for gas atomization represent an average of the fine and coarse microstructures in the powders. Partly by reducing the melt superheat, a du-

Fig. 9. Comparison of Al–Ni–Fe powders atomized using different processes [37].

Fig. 10. Comparison of microstructure of spherical bronze (Cu–13 wt.% Sn) obtained using horizontal gas atomization and IA with varying melt superheats.
plex type of structure was obtained with IA as shown in Fig. 5. The average powder microstructure was quantitatively similar to the gas atomized powders. In Fig. 10 is also seen a comparison between the coarse and fine microstructures in the IA-955 °C melt samples. The fine microstructure in the 955°C atomized IA sample is very similar to the 1237 °C IA sample; while the coarser microstructure in the 955 °C IA sample is somewhat coarser than the gas atomized sample. These results seem to suggest that under similar melt conditions, IA powders experience a higher rate of heat removal by the gas despite the fast moving gas in gas atomization processes.

In a recent study using Cu–6Sn, the microstructure of particles of similar sizes were compared for gas and IA atomized powders [38,39]. As in the case of Al–Fe–Ni alloy and the phosphorous bronze, IA powders of the same size exhibited a finer microstructure. A calorimetric analysis of droplets in gas atomization coupled with an analysis of droplet cooling in both processes, clearly demonstrated that in gas atomization the gas temperature is higher than in IA. This results in a lower driving force for heat transfer in gas atomization resulting in a coarser microstructure. These results were confirmed using a thermal Stokes number, St, calculation [39,40]:

\[
St = \frac{\tau_T \cdot v}{L}
\]

where \( L \) is the length of a cube containing \( mL^3 \) particles (\( m \) is the number of particles per unit volume), \( v \) is the velocity of the gas and \( \tau_T \) is the thermal response time given by:

\[
\tau_T = \frac{\rho_\text{c} \cdot C_\text{p} \cdot D^2}{6k_\text{G}}
\]

where the density, \( \rho \), the specific heat capacity, \( C_\text{p} \), and the diameter, \( D \), are of the particles; while the thermal conductivity, \( k_\text{G} \), is of the gas. A lower St is indicative of increased two way thermal coupling and would result in a larger gas temperature. For gas atomization using a gas to metal mass ratio of 0.95 and 1.35, St was determined to be 1.36 and 2.18, respectively. While for IA, St was calculated to be 8.02. This is in line with the calorimetric study of droplets in gas atomization, the measured gas temperature in both processes and the thermal calculation of droplet solidification.

6. Conclusions

The IA technique is flexible in atomizing a wide range of materials, from Pb to steel, with a controlled size distribution.

Mass flow rate data clearly showed that the throughput from IA is competitive with gas atomization. The gas-droplet heat transfer coefficient may be calculated using the Whitaker equation and the thermal conductivity of the gas evaluated at the droplet surface temperature. This provides a fundamentally correct approach to incorporating the high temperature effects on both the velocity and the thermal boundary layers of the droplet.

Droplet heat loss is controlled by conduction and convection to the gas, the type of gas used in an atomization chamber and the droplet diameter.

Reliable correlations of cell spacing versus cooling rate may be determined using IA. In addition, IA offers a unique tool to carry out fundamental investigations of solidification processing due to its controlled droplet generation, small droplet sizes and reproducible droplet thermal history.

IA powder microstructures were compared with powders atomized using other processes. IA provides conditions for higher heat transfer rates between droplet and gas than is obtained by gas atomization. The thermal Stokes number, St, provides a good analysis of two way thermal coupling between gas and melt spray. St, droplet calorimetry, gas temperature measurements confirmed that two way thermal coupling is greater in gas atomization than in IA, with the former process yielding lower droplet solidification cooling rates.

Acknowledgements

The author would like to thank all members in the Advanced Materials and Processing Laboratory over the past decade who have contributed to this research and carried out many of the experiments and analyzed the results. Financial support from NSERC through the Research Grant and Strategic Projects Programs is gratefully acknowledged.

Appendix A. Nomenclature

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>constant in cell spacing versus cooling rate Eq. (1) determined from a best fit analysis</td>
</tr>
<tr>
<td>Cp</td>
<td>specific heat capacity of the particle</td>
</tr>
<tr>
<td>D</td>
<td>particle diameter</td>
</tr>
<tr>
<td>( k_\text{G} )</td>
<td>thermal conductivity of the gas</td>
</tr>
<tr>
<td>( L )</td>
<td>length of a cube containing ( mL^3 ) particles</td>
</tr>
<tr>
<td>( m )</td>
<td>number of particles per unit volume</td>
</tr>
<tr>
<td>( n )</td>
<td>exponent in cell spacing versus cooling rate Eq. (1) determined from a best fit analysis</td>
</tr>
<tr>
<td>St</td>
<td>thermal Stokes number</td>
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<tr>
<td>( v )</td>
<td>gas velocity</td>
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Greek letters

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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</thead>
<tbody>
<tr>
<td>( \varepsilon )</td>
<td>thermal cooling rate during solidification</td>
</tr>
<tr>
<td>( \lambda )</td>
<td>cell or dendrite arm spacing</td>
</tr>
</tbody>
</table>
References


ρ: particle density
τ: thermal response time