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1	In situ Melt Temperature Analysis During High Pressure Gas Atomization
2	of Liquid Metals
3	
4	A.M. Mullis <sup>*</sup>
5	University of Leeds, Leeds, LS2 9JT, UK.
6	
7	Abstract
8	We explore the extent to which imaging of the melt plume during High Pressure Gas
9	Atomisation using consumer DSLR (Digital Single Lens Reflex) equipment provides useful
10	information about the process. We show that the colour imaging and high spatial resolution
11	can be a useful adjunct to the more widely reported imaging using specialist high frame rate
12	cameras. With knowledge of the camera's colour response curves, the ratio of the signals in
13	the red, green and blue channels can be used to make spatially resolved temperature estimates
14	of the material within the melt plume. Moreover, by combining these temperature estimates,
15	which depend only upon intensity ratios, with the actual intensity of the optical signal we
16	propose it is possible to obtain estimates of the relative surface area of the melt within the
17	plume. This in turn can be related to the local melt fragmentation rate within the atomization
18	plume.
19	
20	Keywords: High pressure gas atomization, DSLR imaging, powder metals, pyrometry.
21	
22	

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## 24 1 Introduction

High Pressure Gas Atomization (HPGA) is one of a number of techniques for producing metal 25 powders direct from the melt, others include water, ultrasonic and centrifugal atomization. 26 However, it is HPGA that is currently receiving most attention due to its ability to produce the 27 28 fine, spherical and satellite free powders demanded by Additive Manufacture (AM), with AM 29 processes driving a near exponential increase in the demand for such powders. Despite this, HPGA remains a relatively poorly understood process, with the complex interaction between 30 31 the melt stream and the supersonic gas jets used to disrupt the melt leading to instabilities on various time scales (for instance, see the review by Anderson & Achelis, 2017). 32

33

One of the factors contributing to this lack of understanding of HPGA is a dearth of techniques 34 to study the atomization process *in situ*. Atomization is usually performed as a batch process 35 with the Particle Size Distribution (PSD) being characterised for the batch of material 36 37 produced, a procedure that does not have the fidelity to capture short timescale variations in the process. A relatively small number of *in situ* investigations have been undertaken using 38 high frame rate video recording and these have significantly improved our understanding of 39 the process, particularly in relation to the unsteady nature of gas atomization. The first such 40 was by Ting et al. (2005) who used Discrete Fourier Transforms (DFT) to analyse high speed 41 video data of the gas atomization process, identifying a range of instabilities with frequencies 42 typically in the range 5-50 Hz, with the dominant instability having a frequency around 25 Hz. 43 44 They ascribed these instabilities to unsteady melt flow to the atomization nozzle, a phenomenon termed melt pulsation. High speed imaging coupled with DFT analysis was also 45 used by Mullis et al. (2008), but now with the region of interest confined to a narrow window 46 just below the melt nozzle. They were able to confirm melt pulsation, with frequencies in the 47 range 25-50 Hz, and also found evidence that the melt spray cone actually consisted of 1 or 48

49 more fast rotating jets of melt (typical frequency ~300 Hz), a feature subsequently confirmed 50 (Mullis *et al.*, 2011) using Particle Image Velocimetry (PIV). Similar conclusions were reached 51 by Achelis *et al.* (2007), who observed that the melt cone in a pressure swirl atomizer 52 comprised a hollow cone of multiple melt filaments. More recently, Bigg & Mullis (2020) have 53 used a statistical object tracking method to construct spatially resolved maps of the melt 54 velocity within the spray cone from multiple frames extracted from a high speed video 55 recording.

56

Modelling studies have also been instrumental in elucidating the processes occurring during 57 HPGA and in filling the knowledge gaps left by a lack of *in situ* observational techniques. A 58 59 detailed Computational Fluid Dynamics (CFD) study by Ting & Anderson (2004) established details of the gas flow-field just below the melt nozzle, with this wake region being described 60 61 as either open or closed, depending upon the gas pressure utilised. Here, a closed-wake is terminated by a downstream Mach disk which tends to prevent gas escaping the wake region, 62 while no such Mach disk is observed for an open-wake. Both structures have also been 63 64 observed experimentally by Mates & Settles (2005) using Schlieren imaging, with the transition between the open- and closed-wake states, both of which are stable in gas only flow, 65 leading to a dramatic change in the melt aspiration pressure experienced at the melt nozzle 66 67 (Ting *et al.*, 2002). A number of subsequent studies have picked up this idea of the open- to closed-wake transition, for instance modifying the shape the atomizer geometry to affect a 68 reduction in the pressure above which closure of the wake is observed (Motaman et al. 2015). 69

70

While the flow behaviour in gas-only flow has been modelled by many authors, the difficulty
in modelling two-phase flow and droplet break-up means that this area has received much less
attention. However, with increasing computing power this has started to be addressed more in

recent years, with simulations that model the melt as both a continuous phase (the Euler-Euler 74 or E-E approach) or as a discrete phase (the Euler-Lagrange or E-L approach). The former has 75 been used by Zeoli et al., (2012) using the volume of fluid (VOF) approach coupled with large 76 eddy simulation (LES) techniques, wherein they identify three modes in which the atomiser 77 may operate, which they term nozzle filming, mixed filming with pinch-off, and no-filming. 78 The coupled VOF-LES method has subsequently been adopted by Neumann et al. (2016) to 79 80 study the breakup of a free falling liquid steel jet, while Li & Fritsching (2017) have used the VOF approach coupled with an Eulerian-Lagrangian approach to simulate the PSD of particles 81 82 formed during gas atomization. Recently, Arachchilage et al. (2019) have also used a similar model to investigate the effect of gas pressure on particle size distribution. However, the E-L 83 approach has tended to be somewhat more computational tractability, which has allowed 84 droplet break-up, either by the development of Kelvin-Helmholtz instabilities (Thompson et 85 al., 2016, Kaiser et al., 2018) or via a wave break-up model (Xu et al., 2020) to be incorporated 86 into the models, which can generally simulate much longer atomisation times than E-E type 87 models. 88

89

While the monitoring of HPGA using high-speed filming techniques has provided both useful 90 insights into the physical processes operating, and provided validation data for the types of 91 92 modelling studies described above, the use of such equipment during commercial production 93 is both expensive and intrusive. Consequently, alternative technologies need to be sought. Mullis & Dunkley (2016a, 2016b) have demonstrated that the use of acoustic monitoring holds 94 promise, although more work is required in this area due to the complexity of relating the 95 96 acoustic signals to atomizer performance. In this paper we explore whether imaging using low cost consumer DSLR (Digital Single Lens Reflex) systems has a role to play in the in situ 97 observation of gas atomization. 98

While DSLR cameras lack the high frame rate of specialist high speed video cameras, they 100 benefit from high resolution and colour imaging. Here we explore whether the additional 101 information provided by colour imaging, a feature not usually available in all but the most 102 expensive of high speed cameras, can provide useful insights into the HPGA process. In 103 particular, we explore whether the colour information in DSLR images can be used for 104 105 temperature estimation within the melt plume and whether this can in turn be used to infer the extent of melt break-up in different regions of the plume. The trade-off is the much reduced 106 107 temporal resolution of the DSLR system, with a typical frame repeat rate of 60 fps, compared to the many thousands of frames per second obtained utilising a high speed camera. 108

109

110 The cost and resolution benefits of using DSLR equipment for temperature measurements have been recognised by a number of previous authors (e.g. Jimenez 2020). Applications include 111 determination of sky quality (Fiorentin et al. 2020) and flame temperatures (e.g. Kuhn et al. 112 2011; Aphale & Desjardin, 2019; Sankaranarayanan et al. 2021 – with Table II in this last 113 article giving a more extensive list of such experiments), with this being applied to industrial 114 incineration processes by Zhou et al. (2022). The accuracy of this approach depends upon a 115 number of camera parameters, most notably ISO setting and shutter speed, with Zhou et al. 116 (2022) reporting mean squared errors between 20-95 K<sup>2</sup>, based on a 60 ton incinerator with a 117 118 maximum flame temperature of 1510 K.

119

As discussed above, the DSLR frame rate is comparable to the typical frequencies of the melt flow instabilities inherent in HPGA systems. In contrast to the melt, the high pressure gas flow is generally considered to be highly stable, reflecting the fact that the melt embodies < 1% of the momentum of the gas and 0.02% of the kinetic energy (see Bigg & Mullis, 2020, based upon a gas velocity calculated via isentropic flow theory of Mach 2.8 and an RMS melt velocity
of 12.8 m s<sup>-1</sup>, giving Reynolds numbers for the gas and melt of 63,000 and 3,900 respectively).
Consequently, while it is likely that such instabilities will be randomly sampled by the DSLR
system, tracking the temporal evolution of individual instabilities remains the preserve of true
high speed filming.

129

# 130 2 Experimental Methods

# 131 <u>2.1 Atomization & Imaging Setup</u>

Atomization trials were conducted using a commercial gas atomizer of the annular slit type 132 operating at a gas manifold pressure of 2.4 MPa. The melt nozzle is of the conventional 133 truncated cone type, with a flat 9 mm tip and a 30° apex angle. The central melt feed tube is 5 134 135 mm in diameter, with the liquid metal being drawn into the atomizer under gravity and by the normal aspiration tendency of this atomizer design, wherein the time-averaged melt feed rate 136 is approximately 0.25 kg s<sup>-1</sup>, with the gas flow rate being 0.35 kg s<sup>-1</sup>. The total atomizing time 137 for a typical 200 kg batch is therefore 15-20 minutes. The melt being processed in the case 138 reported here is 316L stainless steel which is subject to a 200 K superheat, wherein the pour 139 temperature is ~1900 K. 140

141

Imaging of the melt plume was by means of a standard consumer Nikon full-frame (i.e. 35 mm charge-coupled device (CCD)) DSLR camera fitted with a Micro Nikkor 70-180 mm (f/4.5-5.6D) lens. The frame size is  $1920 \times 1080$  pixels at a frame rate of 60 fps with a shutter speed during filming of 1/10000 s. A total of 414 frames were recorded over a filming interval of ~ 7 s. As shown schematically in **Figure 1**, the camera was positioned at the atomizer's viewport with a working distance of around 45 cm and looking slightly upwards towards the melt plume, the camera inclination from the horizontal being ~20°. This was necessary as, unfortunately,

the atomizer was not equipped with a viewport directly in line with the melt plume. A typical 149 150 frame, extracted from the movie, is shown in Figure 2, with some extraneous background having been cropped out of the image. Once this is done, the final image size is  $469 \times 775$ 151 pixels, giving a spatial resolution with the camera as setup above of 0.88 mm/pixel. Due to the 152 153 low temporal sampling rate when using a DSLR camera, the shape of the melt plume varies considerably between frames. However, as shown by Mullis et al. (2017), using high speed 154 filming, the average plume shape (shown inset in Figure 1, in this case averaged over 28665 155 frame) is approximately cylindrical, but somewhat pinched in at the location of the expansion 156 waves of the supersonic gas. Given this is the same atomizer as studied here and operating 157 under very similar conditions, we consider that this is a good representation of the time-158 averaged plume shape, albeit this is not evident from the DSLR images (although equally, the 159 average plume shape is not evident from the individual high speed camera frames from which 160 161 it was generated).

162

# 163 <u>2.2 Image Analysis Methods</u>

Pyrometry, both single colour and two colour, is a well-established temperature metrology 164 technique, with such systems generally using a very narrow part of the spectrum (see e.g. 165 166 Jimenez, 2020 for an overview of two colour pyrometry). In contrast, the DSLR has a much wider spectral response designed to mimic the human eye. As such, the proposition here is 167 much closer to developing a DSLR equivalent of the conventional semi-quantitative foundry 168 technique of using a colour chart to gauge temperature. This will, of necessity, not have the 169 temperature accuracy of a dedicated pyrometry system, but the trade-off is the low cost and 170 high spatial resolution (1920 x 1080) characteristic of modern DSLR systems. A further 171 advantage of consumer DSLR equipment is that it is relatively easy to obtain data on the 172 spectral response of the CCD imaging detector from the numerous hobbyist magazines serving 173

this market. **Figure 3** shows the spectral response functions,  $R_R$ ,  $R_G$  and  $R_B$ , for the red, green and blue channels respectively for camera used to obtain **Figure 2**. As only the ratios of these will be utilised they have been normalised such that the peak of the highest curve is equal to 1.

178 Also shown in the figure is the spectral power,  $S_{\lambda}$ , as a function of wavelength,  $\lambda$ , for that part 179 of the blackbody spectrum that falls within the optical response range, 350-700 nm and for two 180 example temperatures likely to be present within the melt plume, namely T = 1750 K and T =181 1500 K. These curves are given by Planck's law:

182

$$S_{\lambda}(\lambda,T) = \frac{2hc^2}{\lambda^5} \left( \frac{1}{\exp\{hc / \lambda k_B T\} - 1} \right)$$
(1)

183

where *h* is Planck's constant, *c* is the speed of light and  $k_B$  is Boltzmann's constant. We now define the power function,  $B_{\lambda,i}(\lambda, T)$ , (i = R, G, B) for the R, G and B channels respectively by: 186

$$B_{\lambda i}(\lambda, T) = S_{\lambda}(\lambda, T) \times R_{i}(\lambda)$$
<sup>(2)</sup>

187

where  $R_i(\lambda)$ , (i = R, G, B) is the spectral response function as plotted in Figure 3. The function  $B_{\lambda,i}(\lambda, T)$ , evaluated at the two temperatures shown in **Figure 3**, namely T = 1500 K and T =190 1750 K, is given in **Figure 4**. Note that the scales differ by one order of magnitude for T =191 1500 K and T = 1750 K. The response (0-255) of each channel within a pixel will then be 192 proportional to integral,  $I_i$ , over wavelength, of the power function,  $B_{\lambda,i}$ , for the light falling on 193 that pixel, where:

$$I_i(T) = \int_{350 \text{ nm}}^{700 \text{ nm}} B_{\lambda,i}(\lambda, T) \, \mathrm{d}\lambda \tag{3}$$

From Equ. (3) we can estimate the relative response of the R, G and B channels in response to illumination by blackbody radiation of a given temperature. For instance, at the example temperatures used in **Figure 4**, the ratio  $I_R/I_G$  is 2.555 at T = 1500 K and 1.997 at T = 1750 K. Moreover, as we are using only a ratio of the power in different channels, we do not need to consider factors such as the emissivity of the sample or the aperture of the optics used, these being, at least to a first approximation, independent of wavelength.

202

203 From the above we can see that we can, in principle, use the ratio of the power in the R, G and 204 B channels to estimate temperature based on colour. With three available channels we have two unique ratios, which we take as  $I_R/I_G$  and  $I_G/I_B$ , with the evaluation being subject to the 205 206 condition that both the numerator and denominator are > 10. For black areas where both the numerator and denominator of a ratio is 0, this is marked as NaN (not a number), which 207 208 prevents these pixels appearing in any of the contour maps. However, as discussed in more detail below, this generally means that only one or other of the ratios is available for any given 209 210 pixel. By performing the calculation described above for a wide range of temperatures we can 211 build up a 'look-up table' of pixel RGB ratios to equivalent blackbody colour temperatures, 212 this being shown graphically in **Figure 5**. In this calculation we use a temperature range of 600 K - 3200 K, with increments of 2 K. Applying this to an image, such as that shown in Figure 213 214 2, we therefore have the basis for obtaining a spatially resolved map of the temperature within a high pressure gas atomisation plume. 215

It may be imagined that there is a two-fold redundancy in the temperature determination with both the  $I_R/I_G$  and  $I_G/I_B$  ratios being used at each pixel. Unfortunately, this is not the case due

to the large difference between  $I_R$  and  $I_B$ . If the image in **Figure 2** is exposed such that no pixels 219 have any channel (R, G or B) saturated, the intensity recorded in the B-channel is, for the 220 majority of the pixels, so low (< 10 on 0-255 scale) that significant discretisation errors will 221 occur. Conversely, increasing the image exposure leads to significant numbers of pixels in the 222 R-channel being saturated. The best compromise appears to be to expose so as to get a good 223 dynamic range, without saturation, in the G-channel. In the brighter areas of the image this 224 225 leads to pixels which are saturated in the R-channel, but a good temperature determination can be made from  $I_G/I_B$ . Conversely, in the darker areas of the image the signal in the B-channel is 226 227 < 10 (0-255 scale), but a good temperature determination can be made from  $I_R/I_G$ .

In fact, only 0.4% of pixels (8800 out of 1920x1080) fall within the exposure range where both the  $I_R/I_G$  and  $I_G/I_B$  ratios can be used to make a temperature determination, although these give temperatures which map well onto each other. Moreover, the final results may be sanity checked against the experimental conditions, wherein temperatures close to the pour temperature are expected close to the nozzle.

233

So far, we have demonstrated that by using the ratio of the channel intensities,  $I_R/I_G$  and  $I_G/I_B$ that we can make an estimate of the melt plume temperature. However, with reference to the standard Stefan-Boltzmann law for the total power, *P*, emitted by a body of surface area *A* with emissivity  $\varepsilon$ :

238

$$P = \varepsilon \sigma A T^4 \tag{4}$$

239

it is clear that with knowledge of both the temperature of the radiating body and the absolute intensity of the emission, it is possible to also deduce the area of the emitting body (here  $\sigma$  is the Stefan Boltzmann constant), wherein the degree to which the melt is being locally

fragmented may be estimated. Of course, when using a DSLR camera we have to take into 243 account a number of other factors as well. The first of these is that the image of the droplet is 244 245 projected onto the flat imaging plane of the CCD, wherein we detected the apparent area of the projection, rather than the true area of the population of 3D droplets. The second factor is that 246 we also have to take account of the spectral response of the CCD. To address the second factor, 247 rather than using the total radiant flux as expressed by Equ. (4), we consider the absolute 248 249 intensity of one of the channels, in this case  $I_G$ . As explained above, for the exposure model adopted here, the green channel is a better choice than either red, which is frequently saturated 250 251 in the high intensity areas of the image, or blue which generally suffers from low intensity in large areas of the image. Specifically, with the temperature now given at each pixel within the 252 image we use Equ. (3) to estimate the intensity of the signal in the green channel,  $I'_{G}$ , for *unit* 253 emitting area. The ratio of  $I'_{G}$  to the actual response of the pixel,  $I_{G}$ , will then give a relative 254 measure of the radiating area contained within that pixel. That is, contouring the ratio  $I_G / I'_G$ 255 will give a measure of the relative surface area of the melt at each pixel, which in turn is related 256 to the particle size distribution within that pixel and consequently the extent of melt 257 fragmentation. As we are not able to directly calibrate the system, that is we do not know the 258 size, and hence surface area, of the droplets contributing to the emission from any given pixel, 259 260 we restrict the analysis given here to an estimation of *relative surface area* within the plume 261 wherein, we do not need to be concerned with the mapping between apparent surface area in the imaging plane to true surface area of the particles, as this is accounted for in the relative 262 scaling, as is the  $\varepsilon < 1$  for the melt (i.e. the approach is valid for grey, not just black bodies). 263 This *relative surface area* will be a function primarily of the particle size distribution within a 264 particular pixel, with finer particles giving a higher apparent surface area. To a lesser extent it 265 will also reflect the volume of melt being imaged within that pixel, although given the almost 266 cylindrical nature observed for the melt cone (see Figure 1 below) we believe this is likely to 267

be a secondary effect. Consequently, the technique gives a mechanism to image where the gas
is interacting most strongly with the melt within the plume and where the local fragmentation
rate is highest.

271

Implicit within this approach is the assumption that the emissivity of the melt within the plume 272 is constant. Over a limited temperature range (~200 K) this has been shown to be approximately 273 true for various steels (Larciprete et al., 2017), while over a larger temperature range (> 1000 274 K) a number of transition metals have been shown to display a modest increase in emissivity 275 with increasing temperature (Sievers, 1979). However, in most cases surface condition (degree 276 of oxidation, cleanliness etc.) has been shown to be a much more significant influence. 277 Consequently, given the dearth of reliable measurements of metal emissivity as a function of 278 temperature, and the variability therein (see e.g. Figure 1 in Felice & Nash, 2013), we believe 279 280 that the assumption of constant emissivity is likely to be no worse than attempting to correct for any potential variation in emissivity. 281

282

## 283 <u>2.3 Frame Selection & Error Estimation</u>

In order to establish which frames within the movie to analyse in detail we have used the 284 technique suggest Mullis et al. (2008) in which the average brightness of the material passing 285 through a narrow window just below the melt nozzle is assessed. The method is given in detail 286 by Mullis et al. so is not reproduced here, the only deviation from their procedure being that 287 we average across all three colour channels. The sampling window used is 5 pixels high and is 288 289 located 50 pixels below the point at which bright melt is first observed at the top of the frame, with this being shown schematically in Figure 2. This value is chosen so as to ensure the 290 sampling window is fully clear of the melt outlet, which due to the camera orientation is 291 observed as an elliptical orifice, the back edge of which is lower in the image than the edge 292

closest to the camera. The back edge of the melt outlet is not visible in all frames, but where it 293 is, this positioning corresponds to the sampling window being 5 pixels below the lowest point 294 295 of the back edge of the outlet. This also locates the sampling window in the "neck" of the melt plume. i.e. the region where the plume is at its narrowest and is not yet strongly interacting 296 with the supersonic gas. As such, to a good approximation, the melt is subject to negligible 297 fragmentation, wherein the melt brightness within this window may be used as a proxy for the 298 299 volume of melt instantaneously at the melt nozzle. Moreover, as the melt nozzle itself is heated (to prevent freezing of the melt in the initially cold ceramic) there is also only marginal cooling 300 301 of the melt below the pour temperature at this location, wherein the accuracy of the optical temperature determination using the above method can be checked against that of a 302 thermocouple sitting just above the nozzle inlet. For the image shown in Figure 2, the average 303 optically determined temperature within the indicated sampling window is 1862 K (with a 304 variation of  $\pm 2$  K within the sampling window), which compares with a contemporaneous 305 thermocouple reading of 1915 K. However, as we are unable to calibrate at other points within 306 the melt plume, we consider that a typical temperature error of 10% might be realistic. Given 307 the 4<sup>th</sup> power dependence of radiant power (& therefore in our calculation apparent surface 308 area) upon temperature, this would equate to a 40% error in surface area due to errors in 309 310 temperature determination. An overall error of a factor of 2 in area might therefore be in order.

311

#### 312 **3 Results**

A temperature map, corresponding to the image shown in **Figure 2**, is given in **Figure 6a**. This is, as far as we are aware, the first time a spatially resolved temperature map of a gas atomisation plume has been obtained. The results appear reasonable with the brightest areas in **Figure 2** appearing as the hottest in the temperature map (note that only ratios of intensities in the RGB channels were used to determine the temperatures, not absolute intensities) and with

temperatures that seem consistent with the experiment performed, that is with a maximum close 318 to the pour temperature of ~ 1900 K, dropping to around 1000 K at the limit of detection. 319 Although, for the sake of brevity not shown, we have also calculated numerically  $\nabla T$  from the 320 temperature values in the map in order to assess the extent to which we may take the 321 temperature as being uniform with a given pixel, with the magnitude of  $\nabla T$  varying from 322 323 around 2.5 K per pixel in the centre of the hot plume (see position A in Figure 6a as being typical) to  $\sim 150$  K per pixel at the margins of the hottest material within the central plume (see 324 325 position B in **Figure 6a** as being typical). Where these large temperature gradients exist, albeit this being a relatively small area fraction of the total image, it must be remembered that the 326 indicate temperature is an avaerge for the pixel and local variation will occur. 327

328

We note that large areas in the interior of the plume have undergone little cooling which 329 suggests melt in these regions are experiencing only minimal fragmentation due to interaction 330 with the high velocity gas, or that heat transfer is limited as the surrounding gas is in thermal 331 equilibrium with the melt. Conversely, material on the margins of the plume appear much 332 333 cooler, suggesting that significant fragmentation is restricted to this region, with the resulting smaller droplets losing heat more rapidly. This is borne out by the map of *relative melt* 334 projection area given in Figure 6b. Each pixel will contain a range of melt particle sizes, 335 together with melt ligaments and more complex structures indicative of break-up, such as 336 sheets and bags (see e.g. Hsaing and Faeth, 1992). These will have a characteristic projection 337 area on the imaging plane of the camera, which is the measure being determined here. 338 Generally, we would expect finer features (i.e. regions where the melt is interacting most 339 strongly with the high velocity gas) to display the largest *relative melt projection area*. 340

341

We note that the regions of the plume with the highest and lowest *relative melt projection area* differ by around a factor of  $10^5$  (despite a scale that runs from 0 to 255 on the green channel used for this determination). Such a large variation in apparent surface area is permitted in the mapping due to the  $T^4$  dependence of P, i.e. a low temperature high P reading maps onto a much larger value of A than a high temperature low P reading, with this range spanning five orders of magnitude).

As described above, we use the brightness in narrow window just below the melt delivery to 349 350 identify key frames to study. As the melt has not interacted strongly with the gas at this point, it will have experienced little cooling or fragmentation. The resulting time-series may thus be 351 regarded as an indicative melt flow rate of the material at the nozzle for each frame, and is 352 given in **Figure 7**. A number of features are evident from the plot including a high frequency 353 variation in intensity which is not fully resolved by the 60 fps imaging and a long period 354 oscillation with a period comparable to the total filming time of ~ 7 seconds. There is also an 355 extreme low intensity excursion (frame 97, shown in more detail in the insert to Figure 7) in 356 which the intensity drops to 36.9 (0-255 scale), less than half of its mean value of 85.5. We 357 also note a second, relatively extreme excursion in Frame 350 in which the mean intensity 358 drops to 56.4. The mean grey level within the sampling period is 85.53 and if this is associated 359 with the time average melt flow of 0.25 kg s<sup>-1</sup> (and on the reasonable assumption of constant 360 gas flow, measures at 0.35 kg s<sup>-1</sup>) the instantaneous gas to metal ratio would vary from 1.007 361 (frame 44, window mean grey level 118.83) to 3.246 (frame 97, window mean grey level 36.89) 362 compared to its time average value of 1.4. 363

364

Based on the data shown in **Figure 7**, we have selected the following frames for further investigation, both to illustrate the applicability of the technique and to elucidate the

<sup>348</sup> 

instabilities present within the gas atomization technique. As being representative of the normal
functioning of the atomizer we have selected frame 44 which has the highest recorded intensity
of 118.8, frame 243 which has one of the lower recoded intensities (excluding the extreme
excursions discussed above) at 68.5 and frame 269 which has an intensity of 85.3, close to the
global mean value. We also explore in more detail the extreme excursion observed in frame
97, examining this and the frames immediately before and after.

373

The set of images corresponding to the 'normal' operation of the atomizer (frame numbers 44, 374 375 243 and 269) is shown in Figure 8, with the corresponding temperature and *relative melt* projection area maps being given in Figures 9 & 10 respectively. Note that in Figure 9, an 376 artefact is visible in the top third of frames (a) and (c), with this being indicated with two black 377 elliptical outlines in part (a). This is due to the bright melt plume being reflected off the steel 378 gas delivery manifold, which can be seen illuminated as a dull red ring in Figure 8, labelled 379 with the word "Manifold" in part (a) of the figure. This artefact is due to the upward inclination 380 of the camera towards the melt plume and could be eliminated if the camera could be aligned 381 horizontally with the melt plume. With reference to Figures 9 & 10 it is clear that the main 382 difference between periods of low flow (parts b) and other times (parts a & c) is that much less 383 of the interior of the plume is occupied by the hot melt that has minimal interaction with the 384 high speed gas stream. Nonetheless, the coolest material, which is therefore interacting most 385 386 strongly with the high speed gas, is confined to outer margins of the atomization plume.

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The set of images centred around the low intensity excursion in frame 97 is shown in **Figure 11**, with the corresponding temperature and *relative melt projection area* maps given in **Figures 12 & 13** respectively. From these figures the extreme nature of this event is apparent. Specifically, in frame 96 (**Figure 11a**) although there is a strong outflow of bright melt from

the nozzle, the spatial extent of the melt plume is shorter than in any of the parts of Figure 8. 392 This is despite the mean intensity of the near nozzle region for frame 96 being 84.2, close to 393 394 the mean value over the filming period. Following this there is an almost complete shut-down in the flow of melt in frame 97 (Figure 11b) with little evidence of bright melt in the image. 395 This is reflected in the corresponding temperature map (Figure 12b) in which the peak 396 temperature inside the plume is now 300-400 K below the pour temperature. The corresponding 397 398 *relative melt projection area* map (Figure 13b) shows that the low projection area melt, which we would conjecture is essentially coarse and unfragmented, which is ubiquitous in the centre 399 400 of the melt plume in the other frames, is absent here. Finally, in frame 98 (Figure 11c) significant bright melt is once again apparent within the melt plume, although the geometry 401 adopted is rather different from that observed in the other frames. Indeed, the morphology of 402 403 the melt plume is similar to that observed during membrane break-up, which is sometimes observed at the beginning of atomization (Anderson 2011). This would be consistent with the 404 almost complete shut-down of melt flow in frame 97 and its restarting in frame 98. The 405 corresponding temperature (Figure 12c) and area (Figure 13c) maps show that this bright sheet 406 of melt is close to the pour temperature and with low relative melt projection area likely 407 remains largely intact during its first 50-60 mm of transit after being discharged from the melt 408 409 nozzle.

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## 411 **4 Discussion**

With reference to **Figure 6b** we observe that the centre of the plume is dominated by melt with low *relative projection area* that interacts only weakly with the supersonic gas, with the higher area material being confined to the margins of the plume. The inference would be that the interior of the melt plume is likely quite coarse, unfragmented melt, with the highest melt fragmentation rate occurring only on the plume margins where there is a much higher shear

being generated. In this respect the results agree well with those found by Bigg & Mullis 417 (2020), in which high frame rate (16,000 fps) imaging was used to produce spatially resolved 418 maps of the velocity of the material in the melt plume for the same atomizer as studied here. 419 They found a strong interaction between the gas and the melt on the margins of the melt plume, 420 with the fastest moving material contained within a relatively narrow shell at the plume 421 422 margins. Conversely, in the centre of the melt plume they determined that the average velocities 423 were less than 10% of those recorded on the plume margins. In particular, they identified a recirculation zone extending some 35 mm (~4 nozzle diameters) downstream of the melt outlet 424 in which the average melt velocity was  $\approx 0$ , indicating that the melt was just as likely to be 425 moving upwards towards the melt nozzle as away from it. In this recirculation zone shear would 426 be expected to be low and significant breakup of the melt would not be encountered. Likewise, 427 428 in this region extending  $\sim 35$  mm downstream immediately below the melt nozzle outlet we observe low surface area, likely corresponding to coarse particles and filaments of melt. We 429 speculate that this relatively narrow zone in which the melt interacts strongly with the fast 430 moving gas is one of the main causes of the notoriously low efficiency of HPGA processes in 431 432 utilising the considerable kinetic energy embodied in the supersonic gas jet to fragment the melt stream. 433

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We note by comparing the two parts of **Figure 6** that there is a very close correspondence between the temperature and *relative melt projection area* maps, with high temperature being associated with low surface area and *vice versa*. Given the field of view which extends only 60 mm or so downstream of the nozzle we believe this is inevitable, with the extent to which the melt is fragmented dictating the subsequent cooling rate. That is, the temperature of the melt in this near plume region is determined almost exclusively by the extent of breakup and hence the area it present through which it may lose heat. We note that on the margins of the plume, unlike location B in Figure 6a, the temperature gradient is typically quite small (< 10 K per</li>
pixel), wherein the variation in temperature across the pixel is negligible relative to the average
temperature of the pixel (~1000-1200 K), i.e. heat is being lost at a uniform rate across layers
of width equivalent to the pixel resolution (0.88 mm).

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It is widely recognized (see e.g. Li & Fritsching, 2017) that the final PSD in gas atomization is established during secondary break-up (i.e. the break-up of droplets into smaller droplets). In the analysis presented here, considering that the melt plume appears to be visible along its whole length, we believe we are capturing both primary and secondary atomization (primary atomization being the break-up of sheets and ligaments into particles), although as individual melt droplets are not resolved it is not possible to distinguish these two processes within this analysis.

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The distribution relative melt projection area, as extracted from Figure 6b, is shown in 455 Figure 14. We conjecture that a pixel presenting a high surface area will contain a large number 456 457 of small particles, whereas one presenting a low surface area will contain a much smaller number of large particles. As such, the histogram in Figure 14 is a pixel count, contains 458 information about the particle size distribution (by pixel), but is not itself such a size 459 distribution. Moreover, the results may also be skewed by the viewing geometry. Specifically, 460 viewing its projection area may lead to an underestimate of the volume of the interior of the 461 melt cone, where the melt is least fragmented leading to the fraction of low area pixels being 462 463 underestimated. Conversely, we can see from the left- and right-hand margins of the plume (as imaged in e.g. Figure 2) that the melt cone is bounded by a thin, cool layer which present a 464 much higher melt projection area. Given the approximate rotational symmetry of the melt cone 465 it is very likely such a layer exists in front of (and behind) the main body of the melt cone, but 466

is obscured by the much hotter, brighter interior of the melt cone. On balance, we suspect the 467 former effect is dominant over the latter. In some previous works (e.g. Aphale & Desjardin, 468 469 2019), multiple cameras have been used to correct for this viewing geometry effect, although in our case this is not possible as the atomizer has only one viewport from which the melt plume 470 is visible. For similar reasons it was not possible to view the melt plume head-on, nor was it 471 possible to apply a perspective correction for this. To do so would have required modifications 472 473 that would have taken a commercial atomizer out of production for an extended period while additional viewports were fitted, 474

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We have also attempted to correlate the data in the histogram with the corresponding 476 temperature map (Figure 6a) to estimate the number of pixels that contain solid material, and 477 478 those that still contain melt and may therefore be undergoing further fragmentation. However, here there is a further complication in that particles in free flight (i.e. melt in the absence on a 479 container) may undergo significant undercooling below the liquidus temperature before 480 solidification is nucleated, potentially by some 100's of Kelvin for the smallest particles (see 481 e.g. Mullis et al. 2020). In Figure 14 we have indicated where the split between solid particles 482 and melt droplets would occur if the onset of solidification occurred at the liquidus temperature, 483 and also given and indication of how this would be shifted if the average undercooling at 484 nucleation were 75 K or 150 K. From this it is clear that melt temperature and projection area 485 486 on the imaging plane are very closely correlated, the most natural explanation for this being that the degree of melt fragmentation is the primary determinant of the melt temperature. 487

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Factor in the rather large errors associated with the current method and the fact that in the DLSR image fragmentation of the visible melt is incomplete (i.e. some material is fragmented and solid, some is still liquid and continuing to undergo fragmentation), we would assert that

492 we have a technique that could be used to evaluate where within the melt plume significant 493 interaction with the high speed gas is occurring, and by inference to compare this between 494 atomizer designs, but that quantitative *in situ* determination of the particle size distribution 495 during atomization would require a much more sophisticated analysis.

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The variability of the atomization process, as revealed by Figure 7, is consistent with that 497 498 found by Mullis et al. (2008), albeit for an atomizer of different scale (10 kg melt batch, 5 mm nozzle) and design (18 discrete jets with 45° apex angle). That fluctuations in the melt flow of 499 the same order are observed in two HPGA systems of such widely different designs is 500 indicative of the ubiquitous nature of melt pulsation during gas atomization and also reflects 501 on the lack of success that atomizer equipment manufacturers have had in eliminating this 502 issue. As the gas flow will be constant, such fluctuations in the melt flow rate lead to significant 503 504 short term variations in the instantaneous Gas-to-Metal Ratio (GMR). Based on the strong correlation between measured PSD and time-averaged GMR it has been speculated in the 505 literature that melt pulsation leads to short time-scale variations in the PSD of the powder being 506 507 produced (Mullis et al., 2013), although there was no direct evidence of this. However, Figure 13 shows now in more detail the effect upon atomization of a low melt flow excursion. Broadly, 508 as can be seen from Figure 13b, the material displaying high *melt projection area* during 509 periods of low-flow is comparable to that produced at other times, but there is much less of the 510 low melt projection area material. Assuming, as seems reasonable that high area is associated 511 with fine particles, this will shift the mean of the PSD towards smaller values. Moreover, this 512 pattern does not appear to be confined to the extreme excursions highlighted in Figures 11-13. 513 With reference to Figure 10 we can see that even during periods of 'normal' operation, 514 significantly less low area material is present when the flow is lower (Figure 10b) relative to 515 when the melt flow is higher (Figure 10a). 516

# 518 **5 Summary and Conclusions**

We have shown how relatively inexpensive consumer DSLR equipment can provide useful 519 complimentary data to the more commonly reported high speed imaging experiments for 520 521 monitoring the melt plume during close-coupled gas atomization. Using only the ratios of the 522 signals in the R, G and B channels, the colour information within the image can be used to obtain spatially resolved, semi-quantitative temperature maps of the melt plume, akin to the 523 524 traditional foundry technique of using colour charts to gauge temperature. By utilising the absolute intensity data recorded by the CCD we can then also estimate the *relative melt* 525 526 projection area responsible for the optical emission, allowing a spatially resolved estimate of the location of melt break-up within the atomization plume to be made. As far as we are aware 527 this is the first time such a determination of the location of break-up within a melt plume has 528 529 been made directly. As such, the system offers a potentially exciting means to evaluate the performance of atomizer design modification *in situ*, using relatively low cost equipment. The 530 value of the analysis is not so much in being able to predict the PSD of the final powder product, 531 which require a much more complex analysis than that offered here, but rather to visualise 532 where finely divided material (and hence break-up) is located within an atomization plume. 533

534

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Figure 1. Schematic diagram showing orientation of camera to the atomization
system and close-up of the melt nozzle & gas manifold. Supersonic gas flow is
delivered from an annular slit type nozzle (a concentric opening located at the top of
the melt nozzle, with the outer surface of the melt nozzle forming the inner surface of
the gas annulus). The melt plume shape is shown schematically and is based upon
that determined by Mullis *et al.* (2018).





Figure 2. Typical DLSR image of the gas atomisation of 316L stainless steel. Also
shown, towards the top of the image, is the window used to optically measure the melt
flow used in the generation of Figure 7, and for temperature calibration against the
melt tundish thermocouple.

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Figure 3. Relative RGB response of the Nikon CCD used to obtain the colour image shown in Figure 2. Also shown are calculated blackbody curves for material at T =1500 K and T = 1750 K (right hand axis).



Figure 4. Relative power in the R, G, B channels of the camera CCD as a function of object temperature, *T*, for a blackbody source. Note that scales for T = 1500 K and *T* = 1750 K differ by one order of magnitude.



**Figure 5.** Ratio of colour response  $I_R/I_G$  and  $I_G/I_B$  as a function of blackbody 661 temperature for the camera used to obtain **Figure 2**.



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**Figure 6.** (a) Estimated temperature map based on colour channel ratios for the atomization plume shown in Figure 1 and (b) the *relative melt projection area*, calculated on the basis of the absolute intensity in the green channel for material at the local temperature shown in part (a). The locations marked "A" and "B" in part (a) of the figure indicate typical regions of minimum and maximum temperature gradients respectively.



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Figure 7. Typical intensity in each frame of the movie calculated by sampling a narrow
window, 5 pixels high, placed immediately below the melt outlet nozzle. Insert shows
detail of an extreme low intensity excursion identified in frame 97.



Figure 8. Three frames from the movie depicting typical operation of the gas atomizer.
(a) frame 44, brightest mean intensity; (b) frame 243 typical of low mean intensity (c)
frame 269, mean intensity close to its time-averaged global value. The portion of the
gas manifold behind the melt plume is visible in most frames in reflected light as a dull
red ring and is labelled in part (a) of the figure.



Figure 9. Temperature maps corresponding to the three images shown in Figure 8. A
temperature artefact arising from reflection off the gas manifold is visible in some
frames and has been highlighted by two black ellipses in part (a) of the figure.



Figure 10. Maps of *relative melt projection area* corresponding to the three imagesshown in Figure 8.



Figure 11. Three frames from the movie illustrating an extreme low intensity excursion by the atomizer in which melt delivery is close to being shut-down. (a) Frame 96, immediately prior to the melt shut-down, (b) frame 97 during near melt shut-sown and (c) frame 98 atomizer recovering normal operation. Note the morphology of the melt in part (c) which is similar to membrane break-up observed at the start of atomization.



**Figure 12.** Temperature maps corresponding to the three images shown in **Figure 11**.



Figure 13. Maps of *relative melt projection area* corresponding to the three images
shown in Figure 11.

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Figure 14. Histogram showing distribution of log<sub>10</sub> of the *relative melt projection area* (based upon pixel count), extracted from Figure 6b. Also shown are those pixel that contain material below the liquidus temperature of the atomized material (correlating with data from Figure 6a), wherein the material would be solid on the assumption of 0 undercooling for nucleation. We also give an indication of how far to the right the solid/liquid transition would move for an assumed nucleation undercooling of 75 K and 150 K.