Chapter 8

Fluidization with Vibrating Baffles

8.1 Introduction

A fluidized bed equipped with a set of baffles (internals) connected to a vibrating machine was designed and built to make use of the segregation principles as discussed in Chapter 5 to bring about dry classification of particles due to density differences. The set-up was aimed specifically to study and classify very fine metal powders falling within the Geldart groups C and A, supplied by Liverpool University.

The baffles were installed to enhance particle segregation in fluidized bed. However, fine particles falling in the Geldart group C particles are very cohesive and difficult to fluidize, and therefore to classify. An improvement of the working of the baffled fluidized bed was made based on the principle of applying shock waves to the bed material by vibrating the baffles to improve fluidizability (Hoffmann, 2000). This chapter will focus on the technical aspect and the analysis of powders using the fluidized bed with vibrating baffles.

8.2 Fluidization and Separation

Although fluidized beds are best known for their good mixing properties, there is a tendency for particles with dissimilar physical properties to segregate as discussed previously in this thesis. This chapter focuses on the role of the fluidized bed as a particle classifier.

As discussed in Chapter 2, the mixing property of powders in fluidized bed depends much on the bed material, the bed will bubble at a sufficiently high aeration rate, and a fraction of the gas will effectively by-pass the bed in the fluidization bubbles (see Figure 8.1). If there is particle inflow and outflow ('continuous' beds), the bed will act as an ideal mixer unless the aeration rate is kept very low. If a mixture of particles is present, the heavier and/or larger ones will tend to sink. The severity of this effect depends on the particle properties and the aeration rate. This can be a problem, since segregation can lead to defluidization in the bottom of the bed, causing costly process downtime.
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Figure 8.1 Fluidized bed. Vibration ($\omega$) is applied to the tie-rods, baffles are sieve-like with an aperture much greater than the size of the fluidized particles (Ritherdon et al., 2002).

Figure 8.2 Geldart's classification of powders according to fluidization properties (Geldart, 1973).

On the other hand, segregation can be utilized if a particulate product can be caused to collect somewhere in the bed, to be withdrawn preferentially. Another use of segregation is for classification of particles, for instance for recycling. The application of fluidized beds in separation is not a new technology (Kunii and Levenspiel, 1991), but the process is still difficult to control and to be made effective when the powders are sticky or when one uses finer and therefore quite cohesive materials as shown in Figure 8.2. In Figure 8.2, the powder in gray zone will be the subject of interest in this Chapter.
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8.3 Fluidization with Baffles

During earlier research in our group, where particle dynamics in fluidized beds was investigated, it was found that the natural tendency for segregation could be enhanced by incorporating a series of sieve-like baffles in the bed (Hartholt et al., 1995 and Hartholt et al., 1997), as indicated in Figure 8.1.

The baffles had a large open area, and an aperture size much larger than the bed particles. Figure 8.3 shows the effect of baffles on the jetsam concentration-profile in a fluidized bed containing a mixture of different-sized glass beads. The phenomenon of segregation is non-linear in nature: once it has started, it will lead to a decrease in bubble activity low in the bed, which in turn further decreases the mixing, enhancing the segregation.

The key mixing process in bubbling fluidized beds is the upward transport of jetsam (particles naturally tending to sink) in the wakes of, or associated with, rising bubbles. The baffles decrease mixing, and thus enhance segregation, by knocking out part of the bubble wakes (Figure 8.4). Research using baffles in binary mixtures has already proven that such baffles boost segregation. For instance, Fig. 8.3 shows the effect of baffles on the concentration profile of jetsam in a 50-50 mixture by volume of glass beads, about 500 and 250 micron in diameter. The more baffles there are, the better is the segregation. A minimum number of baffles is required to get the process started, but once it is started, it is very substantial, in agreement with the comments above about the non-linearity of the effect.

Figure 8.3 The effect of baffles on the concentration profile of jetsam in a 50-50 (vol.) mixture of glass beads, about 500 and 250µm in diameter (Hoffmann, 2000).
A visual observation of the bed by means of X-ray imaging was performed to verify the idea that the key mixing process in bubbling fluidized beds is the upward transportation of jetsam with fluidization bubbles. Also the effect of the baffles was investigated by using X-ray photography of the bubbling bed (van Dijk et al., 1998) as shown in Figure 8.4. Figure 8.4a shows the wake in a fluidization bubble rising from a layer opaque to X-rays into a more transparent layer in the absence of baffles. The dark wake behind the bubble is clearly distinguishable. In Figure 8.4b, a baffle has been installed, and it can be seen that some wake material is left underneath the baffle.

Figure 8.4(a): a fluidization bubble with wake (b): when a baffle is installed, the wake is left under the baffle (van Dijk, 1998).
More experimental results on fluidization with baffles can be found in Dechsiri et al. (2001).

8.4 Fluidization with Vibrating Baffles

Applying vibration to the bed is known to improve fluidizability and to break interparticle bonds even on a small scale. The underlying principle is that the vibrations decrease the bond strength of the bulk material, thereby increasing its ability to flow. The problem with this method is that the shock waves from the vibration only travel some centimeters into the bed before they are attenuated, leaving much of the bed unimproved.

In earlier work, a new principle to cause the shock waves to penetrate the entire bed was developed (Hoffmann, 2000). Vibration was applied to an internal structure spanning the bed, rather than only applying vibration to the bed-containing vessel or to the gas distributor plate. In our experiments we used a baffle-module as shown in Figures 8.1 and 8.5, but any structure can be used. More details can be found in Hoffmann (2000).

8.5 Metallic Powder Separation Using Fluidized Beds with Vibrating Baffles

8.5.1 Aim and Scope

The aim of this work was to assess the potential for dry classification of metal particles according to their density in fluidized beds with vibrating baffles. The work was carried out in cooperation with the University of Liverpool, where the results were analyzed (Ritherdon et al., 2002).

The powders used were batches of mechanically alloyed (MA), iron-based powders provided by the University of Liverpool. They are so-called oxide dispersion strengthened (ODS) FeCrAl based alloys. The ODS-FeCrAl are an increasingly important group of high temperature alloys due to their high resistance to oxidation, and their excellent high temperature mechanical properties together with much lower costs than, for example, the nickel-based super alloys (Wright et al., 1996, Wilson et al., 1978 and Fleetwood, 1980). The applications of such alloys are components such as heat exchanger tubing in advanced, coal-fired power generation cycles, pressure containment vessels, valves, filters and gas and steam turbine parts (Wright et al., 1993 and Fleetwood, 1980).
The aim of this separation work is to remove particles containing inhomogeneities. The problem is that much of ODS-FeCrAl is produced by mechanical alloying of the constituent elements or of master alloys with minor additions, and during the milling process inhomogeneities in the composition of individual powder particles occur. Investigations show that such inhomogeneities may cause increases in particle density and lead to reduced aluminum content and dispersoid-free regions (Fleetwood, 1980). Therefore, the removal of inhomogeneities is desirable to produce a better quality of powders, particularly if it can be performed continuously as part of a production process, such as by our fluidized bed technique.

This fluidized bed technique has successfully been used to separate polypropylene and polyamide powders with densities of 0.90 and 1.15 g/cm³, respectively (Hoffmann, 2000). However, the aims of our work are to assess whether the technique could be successfully applied to the density separation of metallic powders and, thus, whether it could be incorporated into a process for the production of mechanically alloyed powders.

### 8.5.2 Materials, Equipment and Experimental Technique

#### 8.5.2.1 Powder and Powder Mixtures

The experiments can be categorized into four types (Ritherdon et al., 2002):

1. A commercial, MA, FeCrAl-based powder ($\rho$=7.18 g/cm³, mean diameter 75µm) was used alone in a 10kg charge to assess whether these FeCrAl powders were suitable for fluidization in the apparatus described above. After fluidization trials, samples of powder were taken from the top, middle and bottom of the bed for analysis.

2. A bulk powder in this batch was a 10kg of commercial FeCrAl powder and thus was mixed (seeded) with 0.2kg (2wt.%) of a gas-atomized Fe₃Al powder ($\rho$=6.53 g/cm³, mean diameter 83µm) in order to determine whether such a powder might be separated by means of the difference in their densities.

3. A major aspect of this work was to assess whether the fluidized bed separation technique might be applied commercially, particularly in the production of MA metallic powders such as the MA ODS-FeCrAl alloys. In an attempt to simulate a commercial situation, ~8kg of gas-atomized Fe₃Al powder ($\rho$ = 6.53 g/cm³, mean diameter 83µm) was seeded with ~0.16kg (~2wt.%) of ODS-Fe₃Al sieving ($\rho$ = 6.5-6.6 g/cm³, mean diameter >400µm) known to contain aluminum-depleted inhomogeneities. Separation by density was attempted as might be done in a
commercial production process. The separation process was run both with and without internal baffles in order to assess the effect of the baffles on segregation efficiency.

4. A more complicated charge of powder, consisting of seven different gas-atomized powders of similar morphology and size (average diameter 80\(\mu\)m) but a range of densities, was used to test the sensitivity of the separation technique to differences in density when all other parameters are supposedly equal. The bulk powder was 8kg of a FeCu powder. The composition of this and the seed powders used are shown in Table 8.1 below. Samples of powder from the bed could then be removed and analyzed using SEM EDS. (Scanning Electron Microscopy and Energy Dispersive X-Ray Spectroscopy. With EDS, the chemical composition of a very small surface (about 1 micron) can be determined. Using SEM and EDS together, it is possible to study the surface of a particle and find out the chemical composition of any small feature on the surface.) A random selection of particles from within each sample was used for analysis.

The metallic powders for the separation work were supplied by University of Liverpool, details are shown in Table 8.1.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Density [g/cm(^3)]</th>
<th>Mass [kg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-13Cu</td>
<td>8.02</td>
<td>8.0</td>
</tr>
<tr>
<td>Cu-10.3Al-5.6Ni-4.5Fe-1.1Mn</td>
<td>8.25</td>
<td>0.2</td>
</tr>
<tr>
<td>Cu</td>
<td>8.96</td>
<td>0.2</td>
</tr>
<tr>
<td>Ni-3.4Si-1.2B-1.1Fe</td>
<td>8.59</td>
<td>0.2</td>
</tr>
<tr>
<td>Cu-0.53Ti</td>
<td>8.93</td>
<td>0.2</td>
</tr>
<tr>
<td>Fe-6.1W-4Cr-3.9Mo-1.9V-0.9C</td>
<td>8.60</td>
<td>0.2</td>
</tr>
<tr>
<td>Al</td>
<td>2.70</td>
<td>0.2</td>
</tr>
</tbody>
</table>

8.5.2.2 Fluidized Bed Apparatus

The experiments were carried out in a glass column of 15cm diameter. The relative humidity of fluidizing air was maintained at approximately 30\%. The baffles consisted of woven wires of 0.65 mm diameter with a stitch of 0.42 cm, giving 71.1\% open area. The baffles were attached to three tie-rods as shown in Figure 8.5. The spacing between the baffles is 0.43 cm. The amplitude and frequency of
the vibration source could be adjusted over the ranges 0.5-5 mm and 15-65 Hz, respectively.

The experimental procedure was as follows: for example, in the fourth experiment, each powder was mixed with the bulk material (Fe-13Cu) in sequence. First we started with bulk powder mixed with aluminum powder and then added another seed powder to the bed and so on. The experiment was performed by using 8 kg of the bulk material and 200 g of each seed powder. The seed powder mass was about 2.44% of the mass of the whole bed.

A typical experiment was started at a high gas velocity at which the bed was well mixed. The baffles were inserted and the gas velocity was reduced to the required velocity. Then the vibration was applied to the system. Every experiment employing the vibrating baffles was performed using the minimum amplitude of 0.5mm and around 15-30Hz for the frequency.

![Baffle construction with 0.43 cm separation between the baffles.](image)

The bed was observed visually, and an assessment was made when the system had reached steady state. The air supply was then cut off suddenly to freeze the powder distribution in the bed, and the baffles were pulled out gently, disturbing the system as little as possible. The sampling method used was based on sectioning the bed by a vacuum technique. For 2.44% weight of seed powder, the segregation layer, when one was present, appeared to be around 1 mm thickness. So basically, samples were taken out in approximately 1-mm layer thickness. The powders were analyzed by the University of Liverpool.
8.5.3 Results and Discussion

8.5.3.1 Fluidization of Commercial FeCrAl Powder.

Fluidization of the commercial FeCrAl powder was readily achieved using the technique described above. The predictive equation of Wen and Yu gives a value for the minimum superficial gas velocity, $U_{mf}$, of 0.017 m/s for this system, assuming a material density 8.1 g/cm$^3$ and particle diameter 80 µm (Wen et al., 1966). This $U_{mf}$ value is significantly lower than that needed in previous work with ceramic and polymer powders and bodes well for the incorporation of fluidized beds into metallic powder production processes (Hartholt et al., 1997 and Hoffmann, 2000).

Further tests for the fluidizability of metallic powders-FeCrAl were then performed. They showed that the bed was well fluidized and the large particles in the powder charge sank to the bottom of the bed and smaller particles rose to the top, as can be clearly seen in the optical silhouette micrographs in Figures 8.6a-c.

![Figure 8.6 Optical silhouette micrographs of samples of FeCrAl power taken from the (a) top, (b) middle and (c) bottom of the fluidized bed after fluidization.](image)

8.5.3.2 Separation of two FeCrAl Powders by Density.

Separation of the commercial FeCrAl alloy powder from the gas-atomized Fe$_3$Al powder proved unsuccessful. Samples taken from the top, middle and bottom of the bed after fluidization showed similar levels of the Fe$_3$Al powder evenly mixed throughout the FeCrAl despite extensive adjustment of the fluidization parameters. Although the densities of the two powders differ by almost 10%, and might therefore be expected to separate, the size and morphology of the particles also differed considerably (see Figure 8.7). The effect that this latter difference might
have had on fluidization and separation is not yet fully understood, although it should be noted that if the lighter particles (Fe₃Al) were larger and more spherical than the denser (FeCrAl) ones and that this would have detracted from the effect of the difference in density.

Figure 8.7 SEM micrograph showing the difference in size and morphology of the FeCrAl and Fe₃Al particles used in powder charge type (ii).

8.5.3.3 Effectiveness of the Technique in a Pseudo-Commercial Situation.

Separation of the defective ODS-Fe₃Al seed powder from the gas-atomized Fe₃Al powder was successfully achieved with the seed powder segregating to the bottom of the bed as shown in Figure 8.8. Here 98.5% of the mass of the seed powder was concentrated in the bottom 6mm of the bed. However, the seed particles are of a different morphology than that of the bulk powder and, more importantly, are larger and more massive. All else being equal, larger particles tend to sink in a fluidized bed, so it was necessary to ascertain the degree to which the sinking effect could be influenced or ‘tuned’ by the inclusion of vibrating baffles, rather than simply whether separation was feasible. It was found that removal of the vibrating internal baffles appeared to have only a marginal effect on the separation characteristics of the bed. This is illustrated in Figure 8.9 where the segregation profile of the seed powder is almost identical to that seen in Figure 8.8 with 95.0% of the mass concentrated in the bottom 6mm of the bed. Here only the distribution of the coarse particles is considered relevant. The higher mass% values seen in the bed without baffles are a reflection of differences in the starting concentration of coarse particles and not of segregation efficiency.
It is believed that the effect of particle size is dominant in this system since particle size varies by a factor greater than five while the densities differ only a few percent. However, despite the large difference in particle size and the strong segregation that it elicits, some improvement in the segregation characteristics may be achieved by the inclusion of vibrating baffles.

![Figure 8.8 Concentration of coarse seed particles at different heights in a fluidized bed with internal baffles](image)

![Figure 8.9 Concentration of coarse seed particles at different heights in a fluidized bed without baffles.](image)

8.5.3.4 Sensitivity of the Technique to Density Differences.

After fluidization and freezing of the bed, a segregated layer was visible at the top of the powder. Once removed to a depth of 2mm, this topmost layer was analyzed using a random sample of 100 particles and proved to consist of 93±1vol.% Al. A second layer removed from the 2mm immediately below the first layer contained 35±1vol.% Al. Samples from the center and bottom of the bed contained no
detectable Al particles. It can be seen that segregation of Al powder to the top of the bed had been achieved extremely efficiently and, due to the homogeneity of powder size and morphology throughout the bed, this was achieved purely on the basis of its low density. Only negligible amounts of Al remained below the top 5mm of the powder bed. The densities of the Al powder and the FeCu bulk powder differ by a factor of almost three, favoring the segregation process considerably.

The powder particle type in the remaining mixture with the greatest density difference compared to the bulk FeCu was the Cu powder. In this case the densities differ by a factor of only 1.1. The Cu powder is the denser of the two and ought to segregate to the bottom of the bed. On analysis, this proved to be the case but the results were marginal even with a sample size of 200 particles. A ~2mm thick layer from the bottom of the bed contained 1.5±0.5vol.% Cu in contrast to 0.5±0.5vol.% found at the top of the bed. It would appear that segregation had, indeed, taken place to some extent, but it should be noted that the amount of Cu powder found in powder supposedly enriched in Cu lies below the 2.2vol.% that was added to the bed in the first place. Therefore, at this stage, it cannot be firmly concluded that segregation has taken place, although there is an indication that it has.

Powder particles with densities still closer to that of the FeCu powder showed no segregation due to the fluidized bed technique. The density difference required for segregation can therefore be said to be at least a factor of 1.1 if the Cu powder results are to be believed, but may need to be closer to a factor of three if highly efficient segregation is to be achieved. Efficient segregation was achieved in a polymeric system with densities differing by a factor of 1.3 (Hartholt et al., 1997). At this stage, it appears that separation of metallic powders does not occur as readily as in other systems. We think this problem was caused by differences in particle size and morphology. Further tests with powders of densities differing from that of the bulk by factors between 1.1 and 3 would refine the estimate of the lowest difference required.

8.6 Conclusions

The principle of this technology is to separate powders by means of differences in density. The heavier particle fraction (the jetsam) tends to sink down; on the other hand the lighter particle fraction (flotsam) tends to deposit on the top of the bed. From Table 8.1, the bulk powder (Fe13Cu) has a density around 8 g/cm³. In the first experiment with aluminum, which differs in density from the bulk powder with about a factor of 3-4, most of aluminum could visually be seen to be deposited on the top of the bed after the experiment was performed. This was then vacuumed.
out as a thin layer of 1 mm. This result proved that the technology is capable of separating this type of powder based on a difference in density.

The densities of the rest of the seed powders are very close to the density of bulk powder. As mentioned, this technique is based on the powder’s density differences alone. The separation observed is not very impressive for the mixtures with quite similar densities. The heaviest seed powder, which has a difference in density by a factor of 1.12 did show some segregation but not yet to a satisfactory degree.

Separation in binary mixtures with only small density differences has been shown to be possible in the past. Two plastic powders, polypropylene and polyamide, with densities of 903 kg/m$^3$ 1145 kg/m$^3$, respectively, have been classified in a baffled fluidized bed (Hoffmann, 2000). It therefore remains to be seen what is the lower limits of density difference with these metal powders.

The fluidized bed process is useful, and may well be the most economical of only a few options, for dry classification of mixtures of equal size but of different density. Different size mixtures, on the other hand, can easily be separated by using a simple sieving technique, although, if a continuous process is preferable, and/or the particles are friable, classifying particles by a fluidized bed can be an attractive option also for this type of mixture. The baffles have the highest added value for mixtures with an intermediate tendency to segregate. If the mixture is too cohesive to effect separation in a fluidized bed, the use of baffles to separate will not give a useful improvement. In this case, applying vibration is supposed to boost segregation and improve fluidization by breaking inter-particle bonds but to determine optimal conditions requires more experiments.

Further study should be carried out. Below are some suggestions for experiments that could be done to improve the quality of the segregation:

1. Do more experiments to find the optimal condition for each batch of metal powder by varying the amplitude and frequency of the vibration unit (we think that vibration with a lower amplitude and a higher frequency might be better).

2. Improve the sampling quality, since we cannot vacuum a layer thinner than 1 mm, approximately. One possibility is to remove a relatively thick layer from the larger bed that we are using now, and then treat this sample again in a baffled bed of smaller cross-section. In this way we will get a thicker layer of seed powder in the second bed, and the vacuum sectioning would be more accurate.
8.7 References


