ADVANCED PROCESSING OF METALLIC POWDERS FOR FOSSIL ENERGY APPLICATIONS

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ABSTRACT

This project seeks to enhance the control of metal powder production by gas atomization methods to benefit the implementation of several emerging Fossil Energy technologies that utilize metal powders of specific size ranges and types, which are not efficiently produced by industrial powder makers. Further improvements in fundamental understanding and design of high efficiency gas atomization nozzles is directed toward maximizing powder yields in special size classes, including ultrafine (dia. < 10 μm) powders and mid-range (10-75 μm) powders, with reduced standard deviation. Efficient production of such powders can eliminate a major technological barrier to the use of new concepts for fabrication of hydrogen membranes or thermal sprayed coatings of oxidation resistant alloys, for example. To provide a direct route for rapid transfer of the atomization technology improvements, powder production tests were performed in an up-scaled industrial prototype atomization system. Benchmark trials on some Fe-base alloys, including Fe-16Al-2Cr (wt.%) and Fe-12Cr-1Y, intended for Fossil Energy applications were performed initially to provide assurance that significant improvements can be achieved. In addition, some results will be reported on parallel work involving controlled sintering (into thin porous sheets) of ultra-fine spherical alloy powders, exploring their application as support structures for various types of hydrogen membranes.

INTRODUCTION

The introduction of new types of powder alloys to the commercial marketplace is significantly impeded by the uncertainty of powder size distribution control, i.e., the difficulty of "tuning" an atomization process to accommodate increased production in a size range not normally accessed. For example, specific powder size classes are often obtained by extensive size classification of a typical powder yield with its characteristic powder size distribution, using sieves and pneumatic separators to produce the desired fraction. Especially for complex alloy compositions without

additional markets and with strict impurity limits, e.g., alloy powders for fossil energy applications, this conventional practice is quite costly, because of the losses and (possible) contamination from extensive particle size classification operations and the need to inventory or to scrap the remaining powder that is not desired. A better approach is to gain improved control of the energy transfer process involved with liquid metal disintegration by supersonic gas flows to generate liquid droplets only of the desired size range in the atomization spray, which will solidify to powders of the proper size for a specific application.

It is generally agreed that a close coupled atomization (CCA) nozzle has the most potential for increased efficiency[1], but achievement of such a high level of control will require many process innovations. One such innovation is a basic change in the design of the atomization gas flow passages from the conventional continuous annular slit[2] to a linked series of discrete axi-symmetric gas jets to provide enhanced latitude for nozzle design improvements[3], without some of the nozzle assembly uncertainties. For example, if the gas flow pattern, or curtain, is considered to be a uniform thickness (ideally) with an annular slit gas passage, the gas flow curtain with discrete jets can be adjusted to have periodic variations in inter-jet mixing and gas velocity by variation of jet spacing and diameter and of jet design (converging and converging-diverging). Assuming accurate initial machining (and no damage) of the discrete jet holes, each repeat use of a discrete jet nozzle will reproduce the same gas flow pattern, while each repeat use of an annular slit nozzle depends on the accuracy of assembly to ensure a uniform thickness of the gas curtain, critical for uniformity of the gas/melt coupling and the resulting atomization performance. In addition, a discrete jet nozzle has been used with an internally slotted melt feed tube to provide highly efficient coupling of the kinetic energy of the periodically designed gas flow to the segmented (and stabilized) melt flow with increased uniformity[4], as demonstrated by a shift of the resulting particle size distributions from a bimodal to a single mode distribution.

The enhanced production of ultra-fine powders of high temperature alloys with dia. ≤ 10 μm was chosen for an initial objective of this research thrust involving improved powder processing for various fossil energy applications. Such powders can be used for development of porous sintered metallic support structures (in a laminated type of composite) for inorganic (ceramic) hydrogen separation membranes that are used in coal-fired IGCC applications. Since a physical separation principle is the primary mechanism for this type of composite membrane, it is critical to promote permeability in the metallic support structure (through pore contiguity and reduced wall thickness), while reducing pore size to provide sufficient mechanical support for the thin (less than 2 μm), porous (0.5 to 2 nm pore dia.) ceramic membrane layer[5]. With a properly designed porous ceramic layer coupled to the metallic support structure, the composite membrane can exhibit a maximum permeance for hydrogen with a maximum separation factor[5] for other contaminating gases, e.g., COx, NOx, and SOx. A conceptual sketch of a tubular design of the laminated physical separation membrane is given in Figure 1, which shows an external porous tube (intended to be partially sintered spherical alloy powders for this project) to provide mechanical protection for the fragile interior ceramic membrane. The other important characteristics of the porous metallic support tube include high strength at high temperatures, sufficient ductility, and joining compatibility to permit integration into the synthesis gas separationsystem[5].

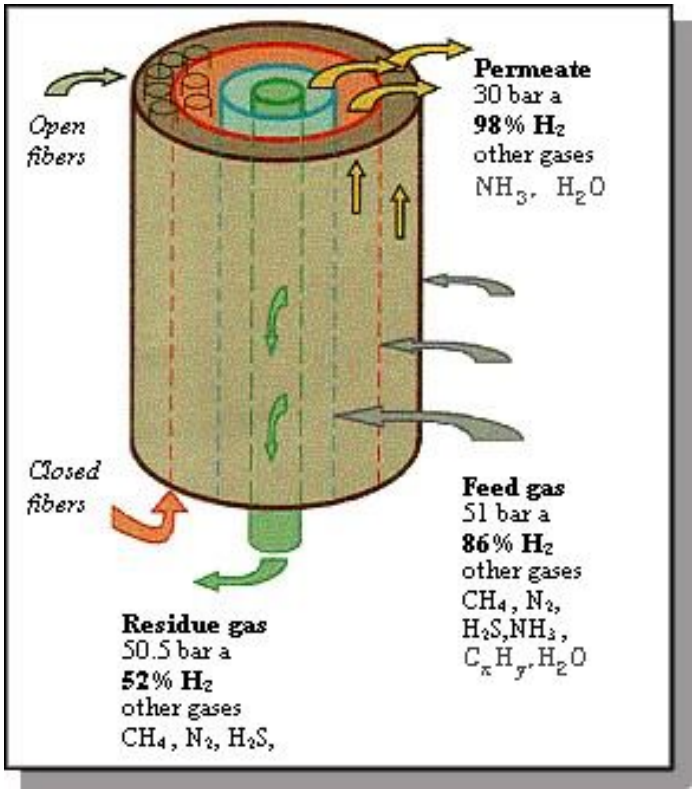


Figure 1. Schematic of a multilayer gas separation membrane (USDOE-FE website).

The requirements for such a porous metallic support layer indicate that spherical ultra-fine metallic powders of high temperature, corrosion resistant alloys would be preferred, but this type of powder is a commercial rarity. One alloy family of current interest for production as ultrafine powder is a simplified iron aluminide composition, Fe-Al-Cr, because of its resistance to integrated gasification-combined cycles (IGCC) environments[6] and compatibility with current ceramic membrane choices.

A second object of the work on this project in the past annual period was concerned with design of an atomization nozzle configuration that would be suitable for production of special alloys for application of protective coatings by thermal spray methods. To pursue this objective, a renewed effort[7] was made to exploit the possible benefits of a discrete jet nozzle with an expanded central bore, as seen in Figure 2. One of the problems that was observed from high speed movies of the previous atomization experiments with this expanded nozzle[7] was an obvious projection of the undisrupted melt stream well into the primary atomization zone before it bent and was atomized in the high shear gas flow on the exterior of the zone. Projection of the melt stream, rather than the intended stream disruption and radial pumping along the slots, was thought to be related to a less stabilized recirculation effect, accentuated by the expanded pour tube orifice area, and to the concave shape of the slotted pour tube, which was less able to cause pre-disruption. Results of experiments with a new pour tube design on an atomization nozzle with a smaller bore[8] indicated that adaptation of the trumpet bell slotted pour tube[9] to the expanded nozzle held promise for promoting melt stream pre-disruption and increased powder size uniformity. In general, the lack

of detailed size distribution analysis results from the previous experiments with the expanded nozzle, all of which had experienced premature melt freeze-off, inhibited a real assessment of its performance. Thus, the latest atomization trial on pure Cu was intended to test the expanded nozzle on a model elemental melt under high melt flow conditions (with an expanded melt orifice), that would have a reduced probability of melt freeze-off and would be more typical of eventual industrial use.

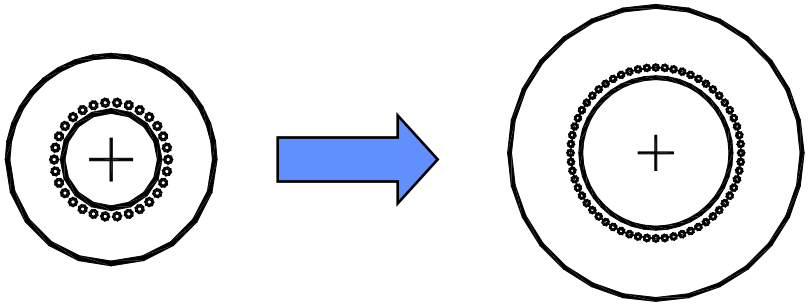


Figure 2. Schematic bottom-view of the enlargement of a nozzle from 30 jets with a 10.4 mm dia. center bore to 60 jets with a 19.5 mm dia. center bore, where the individual jet dia. remains at 0.737 mm.

As described above in connection with the expanded nozzle, one of the key experimental tools for these experiments is observation of the atomization spray origination zone with high-speed (10,000 frame/sec) cinematography at a temporal resolution of 30 ns[10]. If additional nozzle improvements can succeed in forcing the domination of primary disintegration, i.e., in the near-field of the initial gas/melt impact, it is quite possible to gain enhanced control of the droplet disintegration process, resulting in a significant reduction of particle size variance in the resulting powder[10]. Recent progress on comparisons of experimental particle size results to melt disintegration theories suggest that fully expanded gas jet velocity can be used for direct control of particle size[11]. However, further gains in the level of control for gas atomization are needed to verify the operation of the acceleration wave theory[12] that appears to enable particle size control and, thereby, to exploit the obvious advantages for production of special powders. Thus, the atomization experiment with the expanded nozzle and a pure Cu melt was observed with high speed cinematography, as well as being subject to detailed size distribution analysis to compare to a series of previous atomization results on the same pure metal.

EXPERIMENTAL PROCEDURE

A simplified iron aluminide alloy, Fe-16Al-2Cr (wt.%), was used for the up-scaled trials with the Ames Lab industrial prototype high pressure gas atomization (IP-HPGA) system to address the project goals. A schematic of the IP-HPGA system is given in Figure 3 to provide a summary of the features of the system, where several publications can be consulted for details on the operation and its development[10]. Briefly, the full atomization vessel was evacuated to less than 100 milli-torr before backfilling with a full atmosphere of Ar gas to begin melting of the elemental alloy components (99.99%, commercial high purity) in sufficient quantity to make an initial charge

weight of 12 kg for each run. This compares to the use of pre-cast (3.8 kg) Fe-16Al-2Cr (wt.%) ingot for the atomizer charge in the research atomizer[13]. Several attempts were made to melt pre-cast charge in large quantity, but the apparent formation of an oxide shell during the longer (1.5-2X) heating time prevented melt coalescence and fluidity. The molten alloy was poured at 1750˚C and atomized with He gas at a supply pressure of 5.5 or 4.8 MPa (see Table I-A) into a fine droplet spray (see Figure 3) that cooled during free-fall in the spray chamber and solidified into powder which was collected from the powder containers.

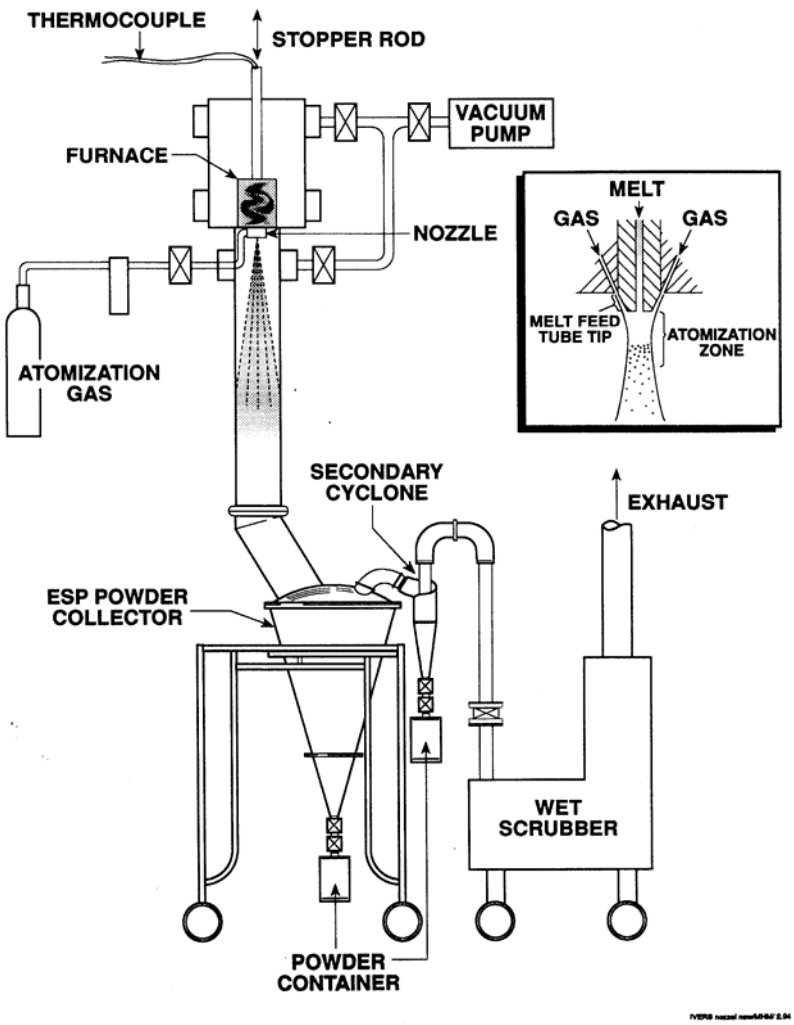


Figure 3. Schematic of the high pressure gas atomization system utilized for powder production in this project. The inset illustrates the essential elements of design and operation of a discrete-jet close-coupled atomization nozzle (in central cross-section), as employed in the system.

Table I-A. Comparison of Starting Processing Parameters

Atomization Run No.	Nozzle (HPGA I type)	Gas Supply Pressure (MPa)	Melt orifice dia. (mm)	Melt orifice profile
BT2-1-149 (Fe-Al-Cr)	45-30-029	5.5	3.2	Flat Base
BT2-1-151 (Fe-Al-Cr)	45-30-029	4.8	3.2	Flat Base
BT2-1-153 (Fe-Al-Cr)	45-30-029	4.8	3.2	Flat Base
GA-1-38 (Cu)	14-60-029	1.2	3.2	Trumpet

To build on the experience of our previous laboratory gas atomization trial[13], Table I-A shows that a prototypical discrete-jet gas atomization nozzle, the HPGA-I (45-30-029), with cylindrical jets, a 45˚ jet apex angle, and 30 jets, each with a dia. of 0.74 mm (0.029”), was employed for He gas atomization. High pressure He atomization gas was selected to exploit the well-accepted advantage (for He relative to Ar or N_2) of a high gas velocity for fine powder production[14]. In the interest of baseline simplification, a standard melt pour tube shape also was utilized, without the slotted internal design that was developed for improved melt filming and atomization uniformity[10].

In the model (Cu) atomization experiment, a trumpet bell slotted pour tube (graphite) was tested against the previous concave slotted pour tube in the expanded nozzle configuration that has been the subject of previous gas atomization attempts[7]. An increased radial spreading distance for the melt stream and a doubling of the gas nozzle exit area was accomplished by use of an HPGA I (14-60-029) nozzle, as listed in Table I-A, with a 14˚ apex angle, a matching (14˚) melt tip angle, and 60 cylindrical discrete jets, each with a diameter of 0.737 mm (0.029 in.), arrayed around a central bore of 19.5 mm, as shown in Fig. 2. A reduced kinetic energy level of the open wake atomization gas (nitrogen) flow was used, where a maximum velocity of Mach 2.2 was employed, based on isentropic free expansion of the cylindrical discrete jets that had a manifold (reservoir) pressure of 1.2 MPa[15]. Corresponding to previous atomization trials for the 14-30-029 nozzle with the concave slotted pour tube[10] and for the 14-22-052 nozzle with the trumpet bell slotted pour tube[8], both under the same velocity conditions and with the same melt orifice of 1.7 mm, 4 kg of pure (99.99%) Cu was melted in a graphite crucible, superheated to 1650˚C, and atomized with ultrahigh purity nitrogen gas. In contrast to the Cu runs mentioned above and the previous attempts with the expanded nozzle[7], an orifice dia. of 3.2 mm was used to provide a sufficient melt stream to reduce the probability of freeze-off.

For the Fe-Al-Cr runs, each process yield was blended and screened at 106µm (ASTM 140 mesh) to eliminate a small amount of atomization debris (splats and irregular agglomerates), as well as at 32µm and 20µm. The weights of these screen size fractions were recorded for a rough estimate of the powder size distribution. A portion of the dia. ≤ 20µm powder was also pneumatically classified in an AccuCut unit (from Donaldson) to dia. < 5µm and dia. < 3µm, with the residual becoming nominally 3-5µm. Test samples of porous metallic sheets were fabricated with a shallow (0.5mm) alumina (99.7%) mold cavity by controlled partial sintering of the ultra-fine spherical Fe-Al-Cr alloy powders in close size fractions (<5µm, <3µm, and 3-5µm), using vacuum sintering at a series of temperatures and times, consistent with the earlier method of hot gas filter fabrication[16]. SEM was used for initial characterization of the sintering results. For the Cu powder run, the complete process yield was blended and screened at 106µm (ASTM 140 mesh) to eliminate a small amount of atomization debris (splats and irregular agglomerates). The resulting powder was riffled to obtain a representative sample for size distribution analysis in an automated laser scattering size analyzer, a Microtrac (from Leeds & Northrup).

RESULTS

Compared to the previous research HPGA system run[13] that was completed in about 1.4 min., the IP-HPGA experiments lasted from 2-4X longer (see Table 1-B), with similar gas (He) and metal (Fe-Al-Cr) mass flow rates. These average flow rates produced a gas/metal mass flow ratio that was at or below 1.0, an excellent value for typical industrial production. The gas volumetric flow rate averaged about 16 m^3/min., which is important to consider for atomization systems with He gas recycling. From the resulting powder a total of about 6.4 kg of dia. < 20µm Fe-16Al-2Cr powder was shipped to Oak Ridge National Lab (ORNL) to support experiments on fabrication of porous membranes for physical separation of hydrogen, as described in the introduction. Also, after additional size classification, about 0.8 kg of dia. < 5µm and about 3.3 kg of 5-20µm of the same powder was also shipped to ORNL.

Table I-B. Comparison of Final Run Data, Fe-Al-Cr Experiments.

Gas flow (kg/min.)	Melt Flow (kg/min.)	Mass Flow ratios (G/M)	Run Time (min.)	Gas Flow (m^3/min.)
2.1	2.1	1.0	5.8	12.5
3.1	3.5	0.9	3.0	18.8
2.8	4.1	0.7	3.4	16.6

A comparison is shown in Fig. 4 of the size distribution analysis of the previous small batch of Fe-Al-Cr powder[13] with the averaged screen size results from the three IP-HPGA runs. Although there is an apparent upward shift of the particle size from the previous run to the present results, this type of shift is typical of the difference between laser scattering size analysis results and screening data. The most significant aspect of the current size results revealed in Fig. 3 is the fact that 30% (by wt. or vol.) of the Fe-Al-Cr powder produced was less than 20µm. Commercial yields of dia. < 20µm powders of such alloys are often far less than 15%, for comparison, typically using either Ar or N_2 atomization gas. As described above, several close size fractions of ultrafine powders were obtained from the dia. <20µm yield for subsequent experiments on partial sintering of porous samples, with size distributions shown in Figure 5.

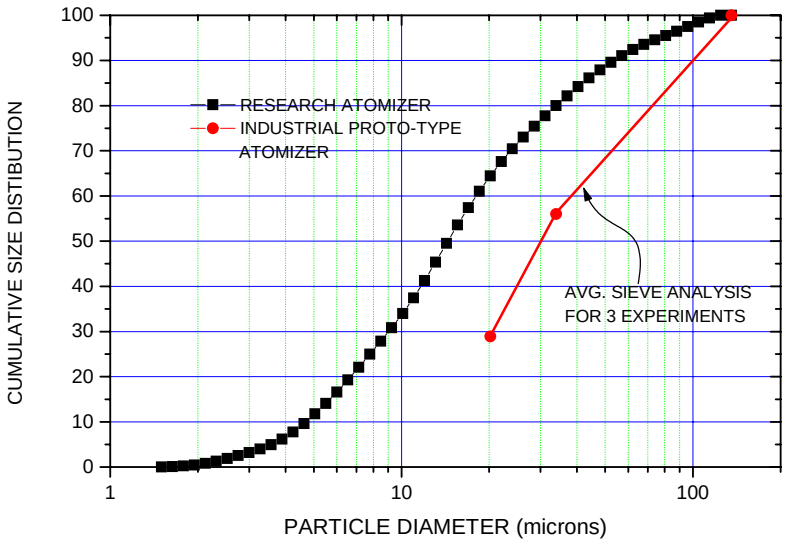


Figure 4. Summary of as-atomized and size classified cumulative size distribution results for He-atomized Fe-16Al-2Cr.

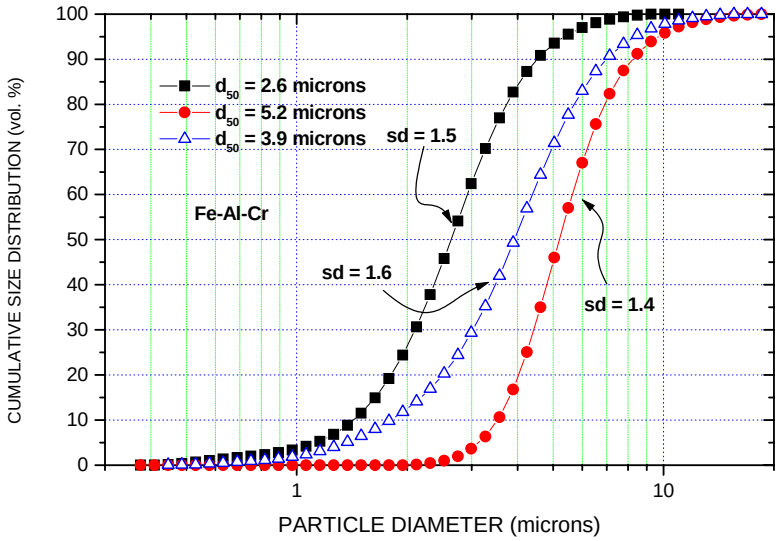
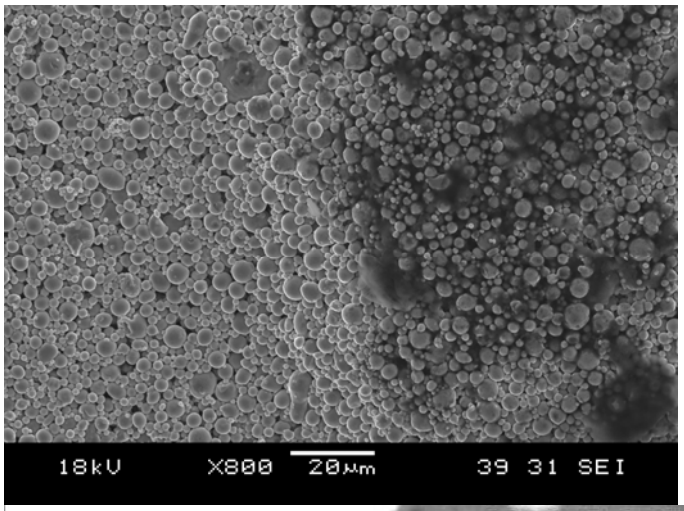


Figure 5. Summary of pneumatically size classified cumulative size distribution results for He-atomized Fe-16Al-2Cr.

The initial experiments on generation of thin porous sheets from the Fe-16Al-2Cr powder involved a seemingly narrow size class, dia. < 5µm. These powders were vacuum sintered at various temperatures from loose powder contained in a 0.5 mm deep alumina mold cavity. A region on the right side of the micrograph in Figure 6a, which shows the sintered surface appearance after 4 h at 1000˚C, appears to be over-sintered. This was verified by a low magnification transmission x-ray radiograph in Fig. 6b that shows several over-densified regions in this thin porous sheet. Previous work[17] has shown that the porous network in such a sample would be partially blocked by the excessive sintered neck growth and particle agglomeration that is evident in Figure 6.

a.)



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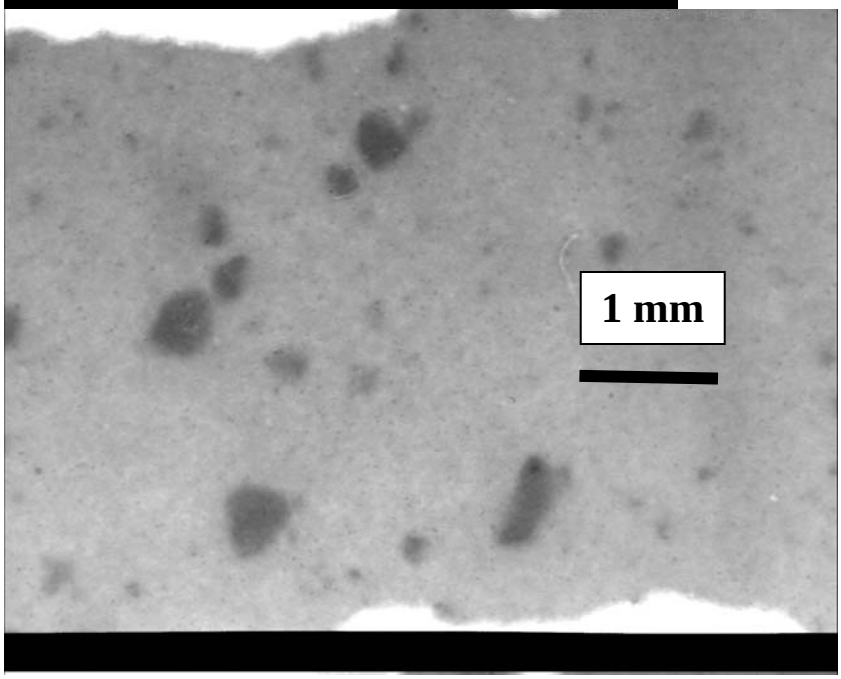

Figure 6. Images of a porous sheet made from Fe-16Al-2Cr powder of the dia. < 5µm size fraction that was vacuum sintered at 1000˚C for 4 h, shown as, a) an SEM micrograph of the sintered surface, and b) a transmission x-ray radiograph.

Thus, it was decided to further subdivide the dia. < 5µm size class to mitigate the apparent problem with widely divergent sintering kinetics of the range of particle sizes in this size class. After size classification, a simple analysis of the resulting size distributions given in Fig. 5 shows that the dia. < 3µm size class has a ratio of largest to smallest powder sizes of significant vol.%, i.e., d90/d10, equal to 2.0, while the d90/d10 ratio of the dia. < 5µm size class equals 3.9. Also, the d90/d10 ratio of the 3-5µm size class is equal to 2.4. The effect of the elimination of the extreme ultrafine powders in the 3-5µm size class is shown in the micrograph of Fig. 7, which shows the sintered surface after sintering at a temperature that is 50˚C higher than the conditions in Fig. 6. Apparently, elimination of any significant number of powders finer than about 3µm was effective at preventing excessive sintering and agglomeration, even after 4 h at 1050˚C; instead promoting the growth of strong necks that are preferred for porous sheet with high integrity[17].

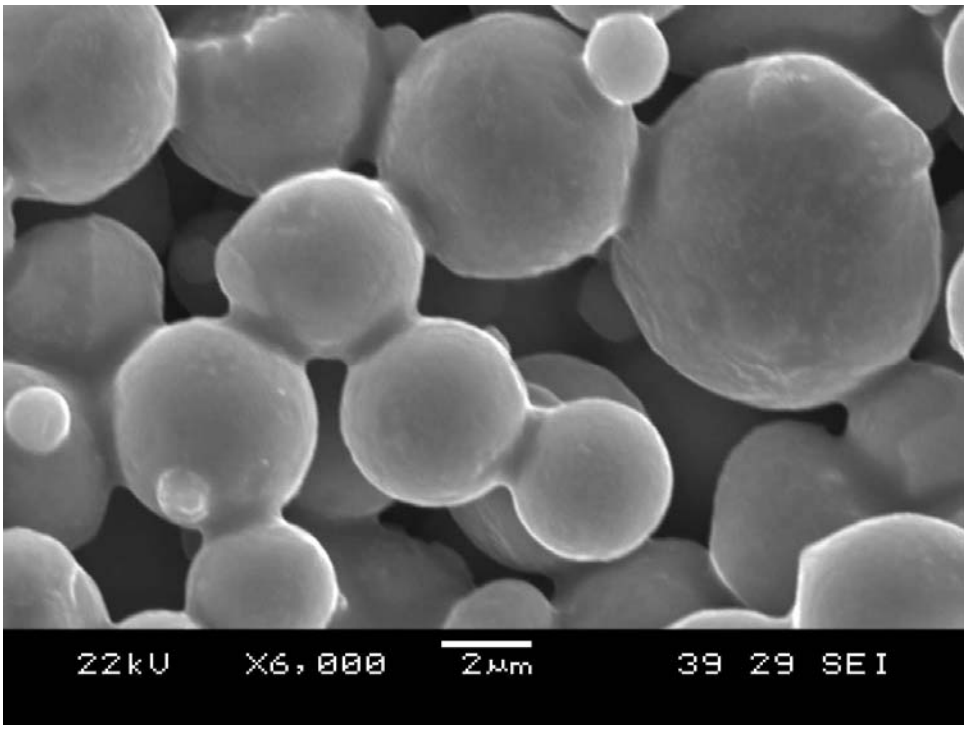

Figure 7. SEM micrograph of the sintered surface of 3-5µm powders of Fe-Al-Cr that were sintered at 1050˚C for 4h.

The gas atomization experiment on Cu with the HPGA I (14-60-029) nozzle was completed in a total time of 44 seconds, with a gas mass flow rate of 10.3 kg/min. and a molten metal mass flow rate of 5.5 kg/min., producing a gas/metal mass flow ratio of 1.9, as given in Table I-C. The gas volumetric flow rate was 8.9 m^3/minute. Size distribution analysis of the resulting powder, as shown in Figure 7, revealed that the d_{50} or average particle dia. (by vol.) is 50 µm. The powder size distribution standard deviation is 1.7, as calculated by d_{84}/d_{50}. The particle size histogram (in vol.%) of these results are compared in Fig. 8 with data from two other Cu atomization experiments; one with the HPGA I (14-30-029) atomization nozzle type, having half of the gas jet holes, and the other with the HPGA I (14-22-052) nozzle, having the same gas jet exit area (as

GA-1-38) by enlargement of each jet diameter[8]. Although all 3 of these experiments used open wake gas flow conditions[11], it should be noted that the current experiment used a melt orifice diameter of 3.2 mm, instead of 1.7 mm, resulting in a significantly increased melt flow (see Table I-A.). Another difference between the parameters is the choice of the interior profile of the melt pour tube, i.e., the current experiment and one of the previous runs used the trumpet bell slotted pour tube[8] and the other previous run used the concave slotted tube. The d50 values of all three runs are significantly different, with the high gas flow and small nozzle run (BT-4-296) exhibiting the smallest powder size, 19µm, and the expanded nozzle run (GA-1-38) displaying the largest powder size, 50µm. However, the standard deviation of the latter is significantly smaller than the other two powder batches, both greater than 2.0 (ref.9). It is also worth noting that the geometric standard deviation (g.s.d.) is 1.8, equal to the smallest value reported for a close-coupled gas atomization process[9].

Table I-C. Comparison of Final Run Data, Cu Experiment.

Gas flow (kg/min.)	Melt flow (kg/min.)	Mass flow ratios (G/M)	d50 (µm)	s.d. (d84/d50)	g.s.d. $(d84/d16)^{1/2}$
10.3	5.5	1.9	50	1.7	1.8

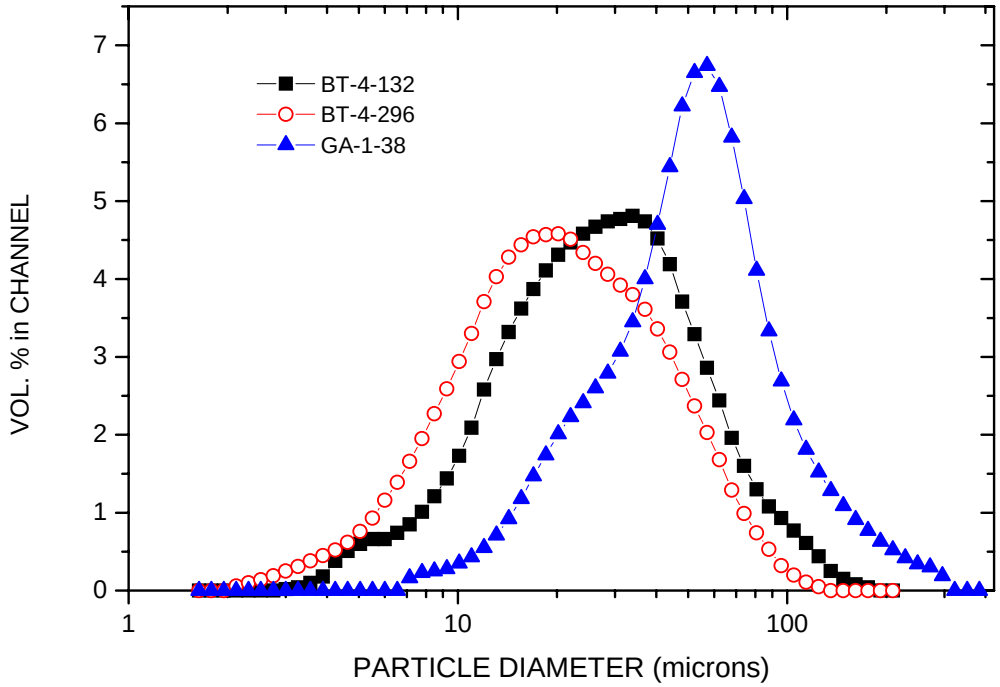

Figure 8. Comparison of particle size histograms for experiments compiled in Table I and other related Cu atomization experiments[9].

DISCUSSION

The high screened yield, 30 wt.%, of fine (dia. < 20 μm) spherical Fe-16Al-2Cr powder, that was generated by discrete-jet, close-coupled gas atomization in the IP-HPGA system with He is an indication of the baseline yield of this type of powder production process. One major reason for the high yield of ultrafine powder is the increased gas velocity of the He, nearly 3 times that of N_2, for example, for the same manifold pressure. According to the acceleration wave model of gas atomization, an increased gas velocity should produce a significant refinement of the average particle size, since the gas velocity (relative to the melt) is a squared factor[12]. While it is true that He is a more expensive gas than, e.g., N_2 or Ar, to utilize for gas atomization, commercial He gas atomization facilities typically employ gas recycling to reduce costs. Moreover, several improvements in the gas atomization process, including modifications in both the gas jets and the melt pour tube design, perhaps with alternative inert atomization gas choices, should enable further increases in the ultrafine powder yield for similar alloys. These improvements in powder availability should be useful for gas separation membrane supports and other significant fossil energy applications.

The powder yields from the three Fe-16Al-2Cr atomization experiments with the IP-HPGA system were classified into several ultrafine powder size fractions to permit preliminary sintering trials for gas membrane support materials. Results of the sintering trials with the broader size class, dia. < 5μm, clearly demonstrated an important issue for fabrication of membrane support structures. To achieve maximum permeability and uniformity of the porous structure, it may be necessary to retain a particle size range that is not significantly wider than about 2-3 particle diameters, i.e., has a d90/d10 ratio between 2 and 3. This will certainly provide additional challenge for powder producers and accentuates the importance of further progress in gas atomization process research to develop more efficient production of ultrafine powders. It is important to note that groups involved with interstitial hydrogen separation membranes are also in need of membrane support layers of similar porosity levels. In addition, there should be a strong benefit for the thin film deposition process (typically for Pd) used in interstitial membranes of a very low surface roughness for the porous support layers[18]. Some ultrafine powders generated from these atomization experiments have already been used to produce porous substrates in support of such research collaborations with Los Alamos National Laboratory (LANL) and further work is planned.

As an important industrial implication of the atomization experiments on Cu with the expanded nozzle, it is certainly desirable that powders produced from expensive alloys for thermal spray applications have a minimal content of dia. < 10μm particles, especially for common plasma arc spraying[1]. These ultrafine powder can cause excessive evaporation and "dusting" during coating application and must be removed, typically by pneumatic size classification. While the current work was performed with Cu, the physical constants of most coating alloys are likely to be similar to Cu (due to their similar melting temperatures) and it may be expected that similar effects on powder size distribution should result from this type of atomization process with coating alloys. The testing of this speculation on coating alloys used for fossil energy applications, for example, will become a part of the on-going work of this overall project to improve gas atomization uniformity and control.

CONCLUSIONS

High screening yields (30%) of fine (dia. <20 μm) spherical powder of a high temperature alloy (Fe-16Al-2Cr) were generated by high pressure gas atomization with He in an industrial prototype atomization system. The atomization gas (He) was selected to provide baseline data on the expected powder size distribution for the use of a typical close-coupled discrete jet nozzle with high gas velocity. The initial powder yield was classified into several ultrafine powder size fractions to permit exploratory sintering trials for gas membrane support materials. Subsequent atomization experiments will be conducted with advanced nozzle designs to explore use of alternative atomization parameters for improved ultrafine powder yields. Powders generated from atomization experiments were used to support research collaborations with other groups in gas membrane fabrication. The unexpectedly narrow particle size distribution of the Cu powder produced with the expanded nozzle bore and increased melt pour rate (with a slotted trumpet bell-shaped pour tube) could be well suited to powder production for thermal spray applications.

ACKNOWLEDGEMENTS

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