

ADVANCED PROCESSING OF METALLIC POWDERS FOR FOSSIL ENERGY APPLICATIONS

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ABSTRACT

This project seeks to develop innovative methods and to enhance process 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 processing. Current improvements in high efficiency gas atomization nozzles were directed toward maximizing powder yields in special size classes, e.g., ultrafine (dia. < 10 μm) powders to eliminate a major barrier to the use of new concepts for fabrication of hydrogen membranes, for example. In the present work, a new Pd thin film membrane support concept was developed that used coarse (40 μm porosity) stainless steel frit to support a thin (75 μm) primary membrane support, made from dia. < 3 μm atomized powder of Fe-16Al-2Cr (wt.%). The membrane support surface, with 0.1-0.5 μm porosity, was sintered at AL and a finished membrane achieved encouraging hydrogen separation performance at LANL. Further sintering process modifications successfully suppressed densification cracking and most sintered surface irregularities, as revealed by SEM on surfaces and cross-sections. Auger electron spectroscopy (AES) with depth profiling analyzed as-sintered and modified surfaces to determine suitability as an inter-diffusion barrier with the Pd-X film. Results of further hydrogen separation work were reported by LANL in a companion paper. The approach of gas atomization with reactive gas (Ar + 5%O₂) for making powders of Fe-Cr (e.g., 410SS) with Y additions was explored further with reactive gas injection (downstream of the breakup zone). The intent is to form an oxide dispersion strengthened (ODS) microstructure upon full consolidation as a viable alternative to mechanical alloying. SEM analysis helped compare as-consolidated Fe-12.5Cr-1Y to a heat-treated microstructure and revealed continued dissolution of Cr₂O₃ at the prior particle boundaries. This is consistent with the desired Y₂O₃ exchange reaction that can strengthen interparticle bonding and promote an ODS microstructure. Hot stage X-ray diffraction tracked the transformations during annealing. TEM analysis of as-HIP samples identified yttria dispersoids of 10-100nm dia. and showed dislocation pinning. Samples were provided to ORNL for additional consolidation testing and characterization.

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 of the desired size range (predominantly) in the atomization spray, which will solidify to powders of the proper size for a specific application¹.

The enhanced production of ultra-fine powders of high temperature alloys with dia. $\leq 10 \mu\text{m}$ was chosen for an initial objective of this research thrust involving improved powder processing for various fossil energy applications. In one important type of application for these special powders, a hydrogen purifying membrane that uses an interstitial separation concept involving rapid atomic diffusion through certain metal lattice structures (e.g., Pt, Pd, V) requires a porous metallic surface with very low surface roughness as a support for effective physical vapor deposition of the continuous metal film². The preferred pore size of the metallic support surface is very small, in the range of 0.1-0.5 μm , to help achieve the desired low surface roughness. To produce controlled porosity of this size range requires ultra-fine powders of high temperature alloys such as Fe-16Al-2Cr (wt.%) with dia. $< 5 \mu\text{m}$. An additional benefit of this alloy choice is the ability to grow (upon annealing in UHP Ar) an alumina scale³ of sufficient thickness to inhibit interdiffusion, especially of Cr and Fe that can otherwise degrade the hydrogen transport rate of the Pd⁴. Another type of hydrogen membrane application, e.g., in a coal-fired IGCC system, uses a physical separation concept with a porous metallic cylinder of an oxidation resistant alloy, e.g., Fe-Cr, as a support for a ceramic membrane with nano-metric pores (5-20 nm) that preferentially pass hydrogen and reject larger gas atoms and molecules, e.g., CO, CO₂, NO_x, and SO_x(5). The pore size that has been found most beneficial for the support cylinder is 1-2 μm , requiring powders of about 10 μm diameter. For further advances into elevated temperatures for this type of membrane, it is also desirable to utilize a type of powder for the structural membrane support that resists creep and continued sintering and pore closure to well above 1000C. Preliminary experiments at ORNL indicated that a novel type of oxide dispersion-strengthened (ODS) stainless steel powder made from Fe-Cr-Y by a reactive gas atomization process at Ames Lab is suitable for this type of application. Such special high temperature alloy powders in ultrafine sizes (dia. $< 10\mu\text{m}$) are either extremely expensive, of insufficient quality, or unavailable commercially.

Previous work on the support structures for hydrogen purification membranes had attempted to fabricate a freestanding thin wafer form of a porous substrate surface from dia. $< 3\mu\text{m}$ powders of Fe-16Al-2Cr (wt.%) powders⁶. These studies achieved a sufficient sintered state in this wafer

structure to exhibit the proper degree of low surface roughness and open porosity⁶. However, a cracking problem related to sintering shrinkage was encountered with the porous wafers, even when supported on a loose bed of high purity alumina particles during sintering. These problems with mechanical integrity made it difficult to deposit a continuous Pd film and to test the full membrane for hydrogen permeation. Thus, advancement of this work demanded a new fabrication procedure for the Pd membrane porous support layer.

A novel processing technique for Initial experiments on generation of a novel type of oxide dispersion-strengthened (ODS) stainless steel powder made from Fe-Cr-Y by a reactive gas atomization process had succeeded in producing only small quantities of powder due to premature termination of the melt stream flow. Promising results were achieved for this powder as a precursor for simplified production of ODS ferritic stainless steel alloys with isotropic microstructure, compared to the conventional mechanical alloying approach that results in anisotropic mechanical properties⁷. These results and the need for porous supports for ceramic membranes encouraged further development of the in situ gas phase reaction process to enable fully successful atomization runs to be completed. Diagnosis of the previous problem indicated that a high local melt viscosity increase from excess oxygen dissolution at the melt pouring orifice due to a close-coupled nozzle recirculation effect¹, which may have lead to premature “freeze-off” of the melt stream. Thus, new efforts were initiated to inject the reactive gas mixture at a location downstream of the atomization nozzle, but where the atomized particles were still hot enough to experience excess surface oxidation. In addition, further characterization of the HIP consolidated microstructure of these special powders was performed to gather more evidence of Y₂O₃ dispersoid formation. Heat treatment effects were also explored by high temperature X-ray diffraction of loose powders and by SEM analysis of consolidated microstructures.

EXPERIMENTAL PROCEDURE

Powders of an iron aluminide alloy, Fe-16Al-2Cr (wt.%), were produced with a high-pressure gas atomization (HPGA) system¹ to use in the current sintering experiments to make porous membrane support surfaces. Briefly, the full atomization vessel was evacuated to less than 100 mtorr 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. The molten alloy was poured at 1700°C and atomized with Ar gas at a supply pressure of 5.5 MPa into a fine droplet spray that cooled during free-fall in the spray chamber and solidified into powder, which was collected from the powder containers¹. The powder yield was blended and pre-screened at 106µm (ASTM 140 mesh) to eliminate a small amount of atomization debris (splats and irregular agglomerates). A full set of ASTM screens with opening sizes below 140 mesh and down to 635 mesh (20µm) were used to obtain an estimate of the particle size distribution¹. A portion of the < 20µm dia. powder was pneumatically classified (AccuCut, Donaldson, Inc.) to < 3µm dia. for the sintering experiments. Analysis of the cracking problems during sintering of the free-standing porous wafers lead to development of a new approach that uses a secondary support frit underneath the membrane support, as shown in Fig. 1, to add some mechanical interlocking and structural stiffness.

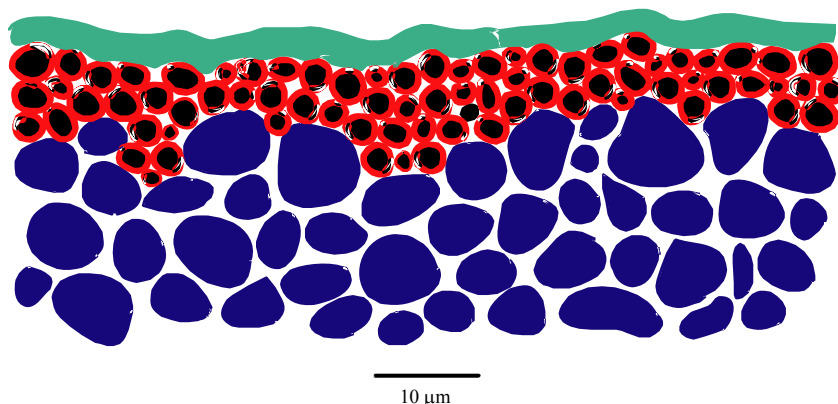


Figure 1. Schematic of a revised approach for more robust support of a Pd interstitial hydrogen separation membrane.

To implement this approach, a sample configuration was designed that included a 9.6 mm O.D. x 6.4 mm I.D. x 2 cm length alloy (Inconel 625) tube that was counter-bored to accept a 316L stainless steel frit (40 μm avg. pore size) that was press fit from one end (into the enlarged bore). The exterior surface of the frit was set into the end face (either flat or chamfered) of the tube by about 75 μm to provide a recessed mold cavity for slurry “casting” of the ultra-fine powder of Fe-16Al-2Cr. In this slurry casting operation, the < 3 μm dia. powder was blended with methanol and poured onto the recessed mold cavity, followed by immediate “striking-off” of the surface with a planar (razor) blade to produce a reasonably flat powder surface. Evaporation of the residual methanol was performed at ambient temperature in air, until the surface appeared dry. Each sample was vacuum (10^{-6} torr) sintered at 975C for times of 1-4 hours, based on previous work⁶. Optical microscopy and SEM of the sintered surface and a mounted and polished cross-section (unetched) of each sample provided initial characterization of the sintering results. Auger electron spectroscopy with depth profiling allowed measurement of the (Al) oxide film thickness in the as-sintered condition and after post-sintering heat treatment (in UHP Ar atmosphere at 800C for 24h) for controlled oxide growth.

New efforts to produce an externally oxidized precursor powder for ODS microstructures utilized 4kg of an Fe-12.5Cr-1.0Y (wt.%) alloy that was atomized in an HPGA system at a pouring temperature of 1700C with UHP Ar gas at a supply pressure of 6.9 MPa with a 45-30-029 HPGA nozzle⁸. A reactive gas mixture, Ar + 5%O₂, was injected (vertically down) through a 20 cm dia. “halo” nozzle at a location 20 cm downstream of the atomization nozzle to interact with the particle spray that was expanding conically during freefall in the spray chamber, as shown in Fig. 2. The resulting powder was collected from the collection cans and chamber and was blended and pre-screened at 106 μm (ASTM 140 mesh) to eliminate a small amount of atomization debris (splats and irregular agglomerates). The resulting sub-106 μm powder was screened at 45 μm to generate < 45 μm powder samples for bulk oxygen analysis (by dissolved gas fusion, LECO unit) and for chemical analysis by ICP-AES by a vendor (NSL Analytical Labs). The balance of the powder was screened at 53 and 20 μm to prepare samples for sintering and hot isostatic pressing (HIP) experiments, similar to the previous work⁹, to examine consolidation processing options.

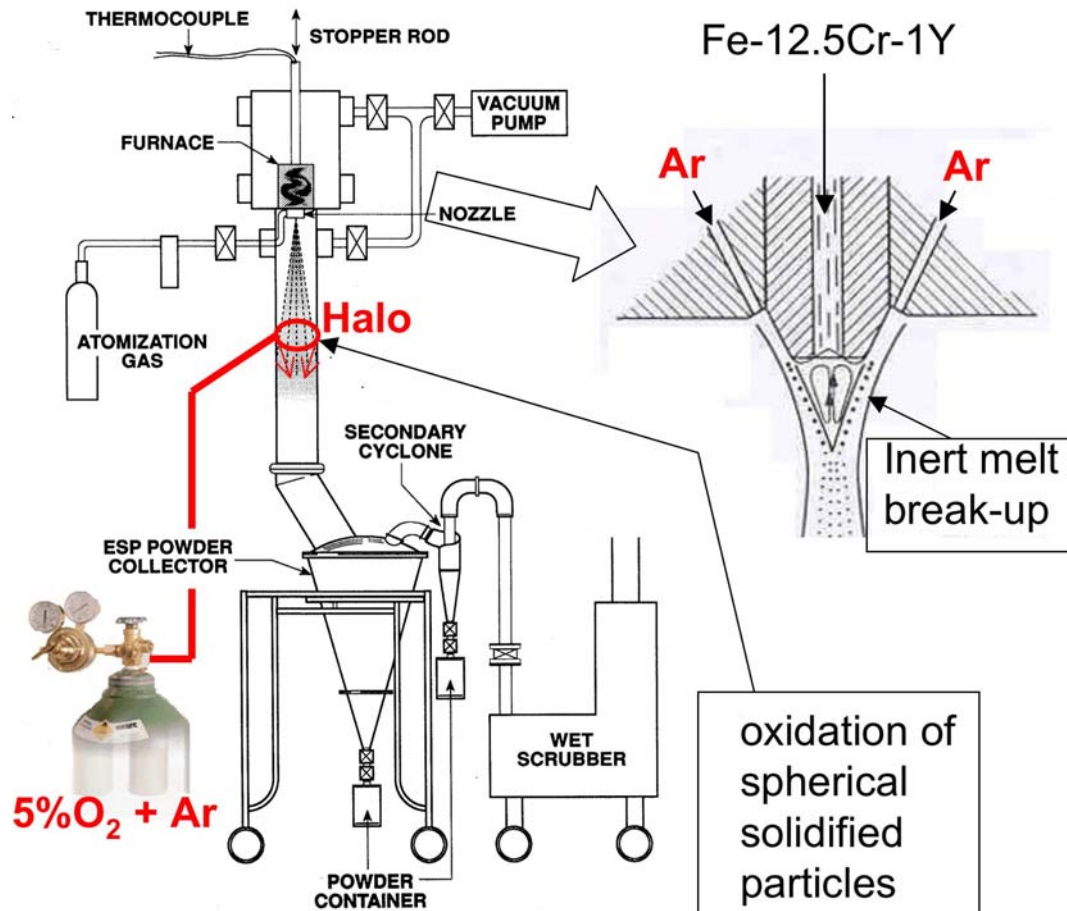


Figure 2. Schematic of downstream halo approach to injection of a reactive gas mixture ($\text{Ar} + 5\% \text{O}_2$) into the HPGA system at a distance of 20 cm below the atomization nozzle (shown in the inset sketch) that was supplied with UHP Ar gas.

In addition, further characterization of as-atomized powder from a previous experiment⁸ was performed to characterize the heat treatment response of loose powder ($< 20 \mu\text{m}$ dia.) that exhibited 2 wt.% oxygen content. Heat treatment effects were explored by preliminary measurements using high temperature (800 to 1200C in 50C increments) X-ray diffraction (Cu K- α radiation) of loose powders in a continuously pumped (sensor not working) vacuum atmosphere using a PANalytical X-Pert Pro diffraction system. Further characterization of HIP consolidated microstructures from previous experiments was performed to gather more evidence of decomposition of the Cr_2O_3 phase on prior particle boundaries by SEM analysis of the microstructures, using quantitative image analysis methods (ImagePro software).

RESULTS AND DISCUSSION

The initial test of the new approach for fabrication of the Pd membrane support produced a fairly promising result, but the surface still exhibited several significant types of defects. The sample configuration started with a flat surface on the projecting end of the tube and the sintering parameters were 4 h at 975C. Analysis of optical microstructures (given in Fig. 3) and SEM

observations indicated that the powder sintering time (4 h) was too long, leading to excess sintering shrinkage stress that promotes centerline and rim cracking. It is useful to note that the stress state generated in middle and at interface with tube interior wall was probably tensile.

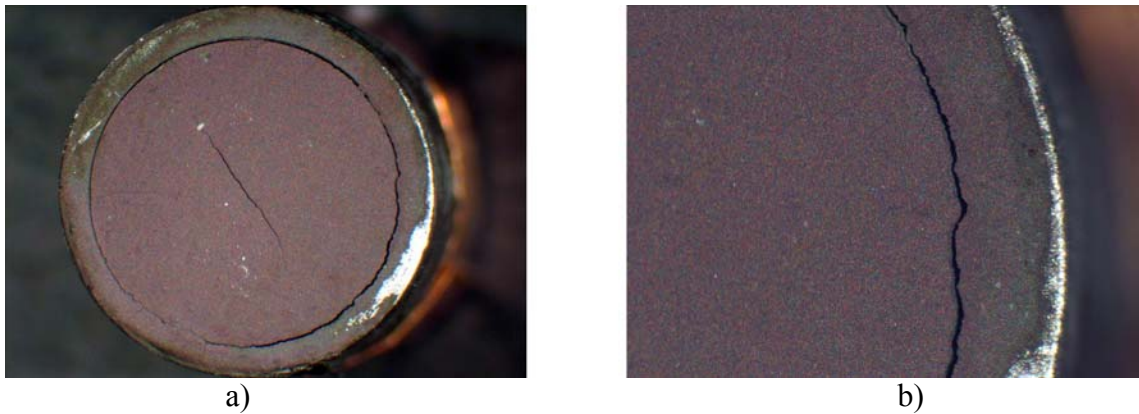


Figure 3. Optical micrographs of two views of the initial sintering experiment, showing a) a significant centerline crack and a clear circumferential crack that extended around the interface with the interior of the tube, and b) a higher magnification of a region of the circumferential crack.

A second experiment was conducted with the same sample configuration, but using a shorter sintering time of 1 h. As Fig. 4 shows, the shorter sintering time was effective apparently at reduction of sintering shrinkage and tensile stresses, sufficient to eliminate the centerline cracking. Unfortunately, evidence of circumferential cracking persisted, leading to the need for a sample configuration change to accommodate the remaining tensile stresses that must have been too high for the thin (75 μm) sintered layer.

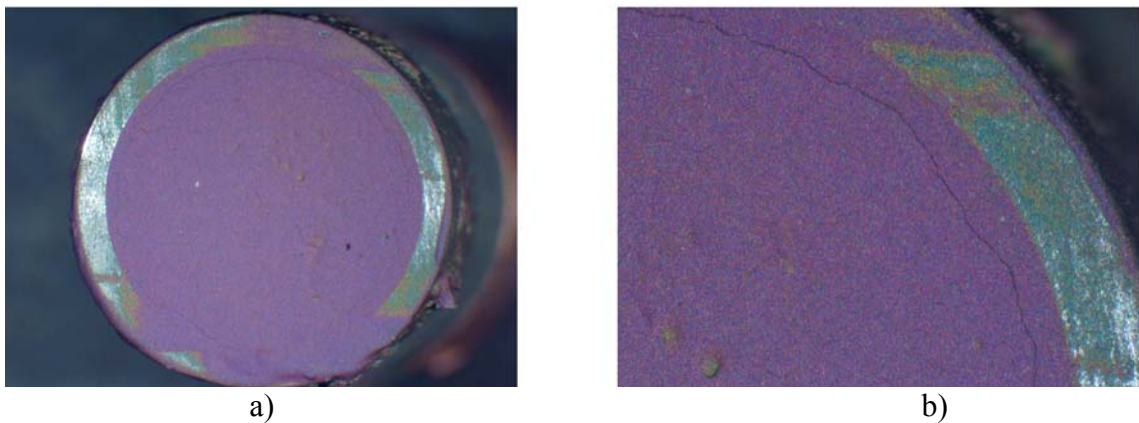


Figure 4. Optical micrographs of two views of the second sintering experiment, showing a) no centerline cracking and a very minor circumferential crack in one region, slightly inward of the interior interface of the tube, and b) a higher magnification of a region of the minor circumferential crack.

One idea for changing the design of the sample configuration involved the cutting of a 3 degree (from horizontal) chamfer in the flat end of the tube (see Fig. 5a) to allow the radial sintering shrinkage to be accommodated by sliding along the chamfered surface without building tensile stresses in the thin layer. The optical micrograph in Fig 5b reveals that this configuration was successful at eliminating the last evidence of circumferential cracking. Also, the SEM

micrograph in Fig 5c shows that the sliding mechanism worked as intended, where a short length of separation appeared along the lower region of the chamfer. However, the majority of this inclined surface was sealed well and this new design feature of the tube seemed to help maintain the full layer in a crack-free condition.

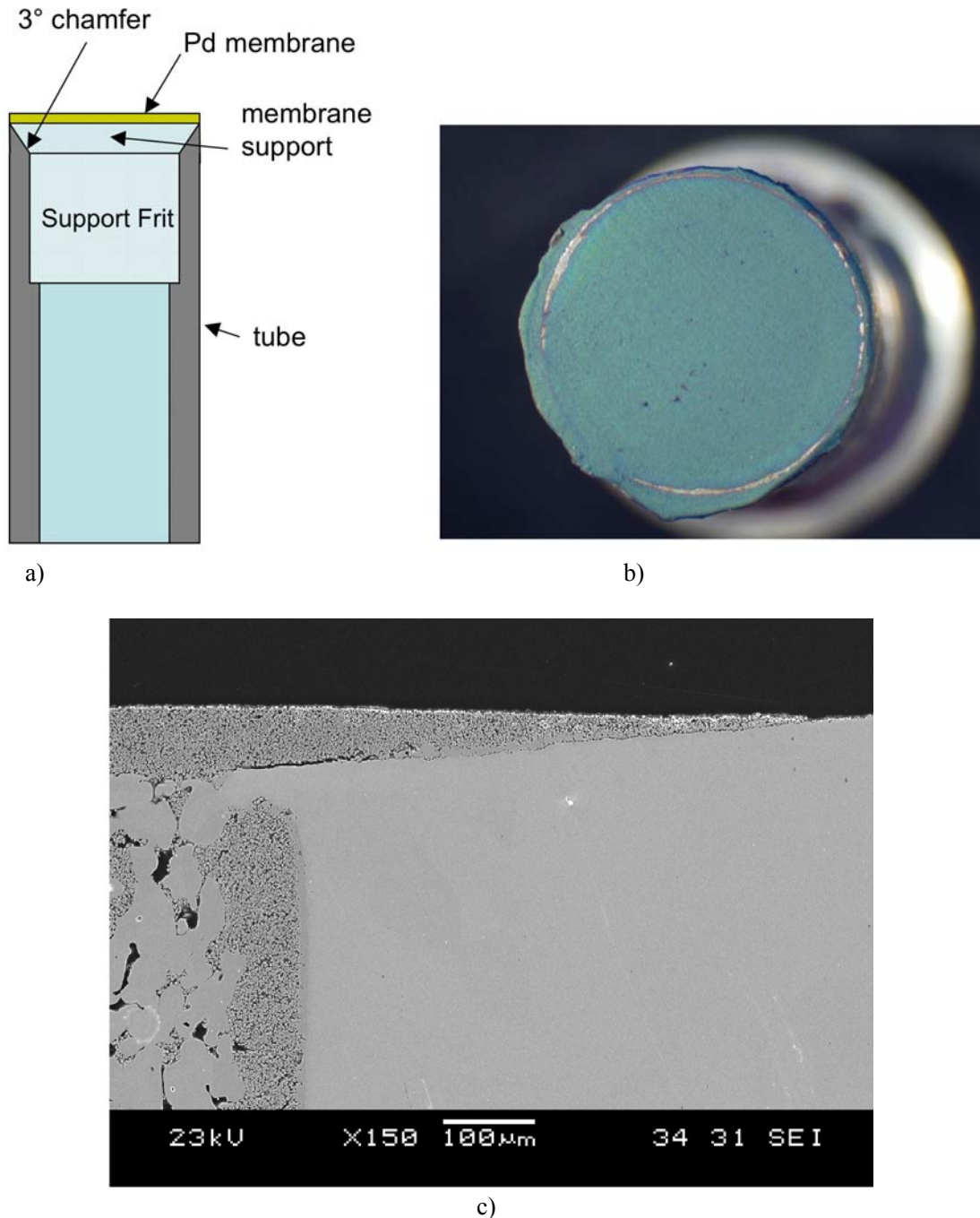


Figure 5. Collection of illustrations to show, a) the chamfered tube configuration (not to scale) with labels on the important features, b) an optical micrograph of the sintered surface (uncracked) of the membrane support layer, and c) a cross-section SEM micrograph of the chamfered region of the membrane support, showing the true angle and the degree of integrity maintained by the sintered layer in the tube interface region.

Closer examination of the sintered surface shown in Fig. 5b with the SEM revealed other types of defects (see Fig. 6a). The most significant defect from the standpoint of subsequent PVD deposition of a Pd thin film appeared to be the type of “sink hole” pit/depression shown in Fig. 6a and, in more detail, in Fig. 6b. It is unlikely that a Pd film could bridge this type of gap, which could produce a “pinhole” defect in the resulting membrane film, an unacceptable condition for high purity hydrogen separation. Thus, the source of these sinkholes needed to be found to enable an approach to be developed to eliminate them from the membrane support. Fortunately, SEM cross-sections (see Figs. 6c and 6d) were able to reveal the general sinkhole source, namely excessive penetration of the ultrafine powders down into the large (40 μ m) frit porosity. Apparently, the methanol carrier fluid assisted powder flow (during slurry application) down into the relatively wide pores (see Fig. 6d) of the stainless steel frit that was selected for this experiment. Following analysis of additional experimental results, it was concluded that the future sample configuration should substitute a stainless steel frit with a significantly finer average pore size, where probably about 10 μ m would be ideal.

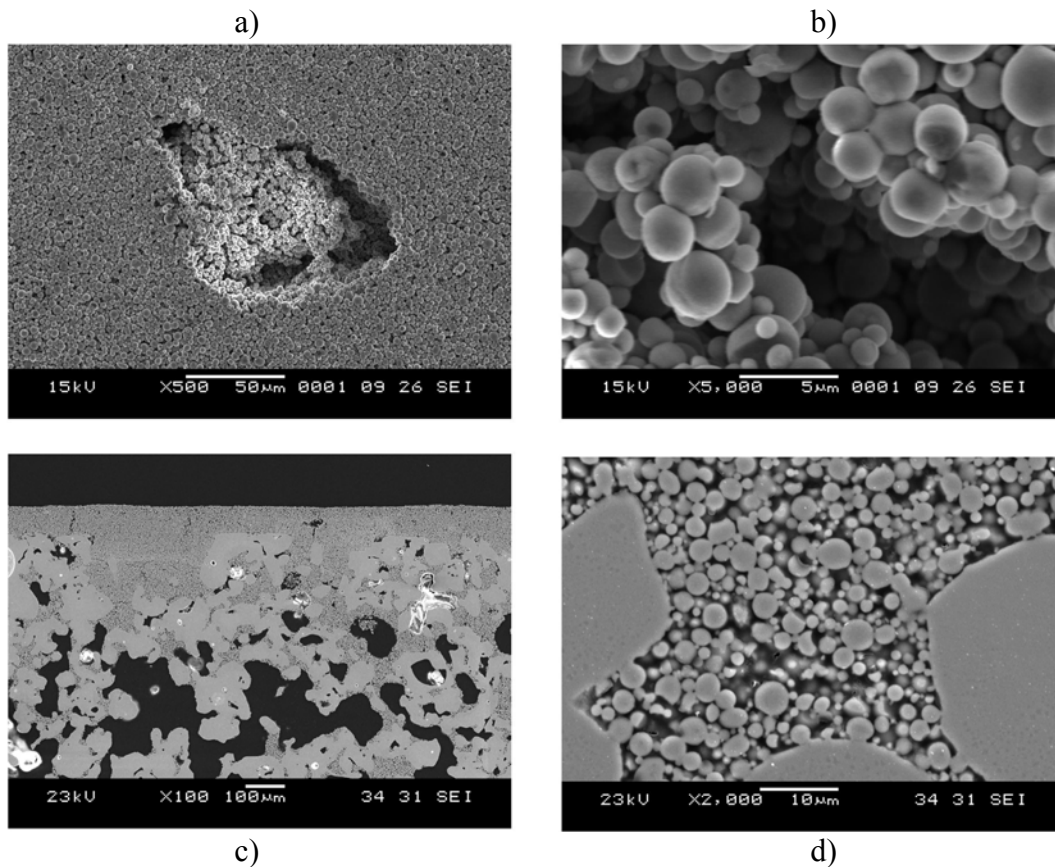


Figure 6. Assembly of SEM micrographs showing, a) a sinkhole type of sintering defect in the membrane support surface of Fig. 5, b) higher magnification of the defect, showing the depth and span of the hole, c) cross-section of the sintered membrane support, revealing the excessive penetration depth of the $< 3 \mu\text{m}$ powder, and d) a higher magnification of the passage between pores in the frit that enabled such flow.

Another important feature of the porous membrane support system that required development was a method to apply a diffusion barrier coating to the exposed surface of the membrane

support, at the interface where the Pd thin film will be deposited. As mentioned in the introduction, the Fe-16Al-2Cr alloy is known to be capable of forming an alumina scale, following a controlled oxidation annealing treatment, i.e., 800C in a UHP Ar atmosphere for 24h (3). The surface of a membrane support sample was examined before this treatment by Auger electron spectroscopy with depth profiling, as shown in Fig. 7a, and the results indicated an Al oxide film thickness of about 5-7 nm. Re-examination of the sample surface after the oxidation anneal revealed an alumina film thickness of about 10-12 nm, as indicated in Fig. 7b, approximately double the as-sintered thickness. While experimental verification of the barrier film coating effectiveness at suppressing interdiffusion into a Pd film remains to be performed, the ability to grow a significant alumina scale on the surface appears promising for this objective.

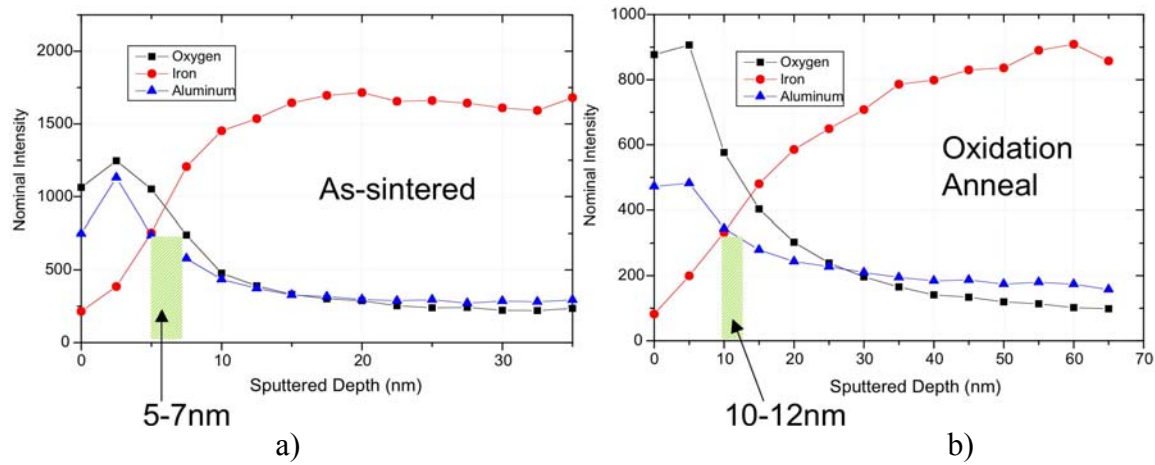


Figure 7. Summary of results of Auger electron spectroscopy at a series of depth profiling increments for, a) the as-sintered surface of a Fe-16Al-2Cr porous membrane support and, b) the same surface after an oxidation anneal.

Previous experiments that produced an externally oxidized precursor powder for ODS microstructures utilized an Fe-12.5Cr-1.0Y (wt.%) alloy that was subject to an in situ gas phase reaction with Ar + 5%O₂ atomization gas¹⁰. To solve the problem with premature melt stream freeze-off from excess oxygen dissolution at the melt pouring orifice, new efforts involved the use of UHP Ar atomization gas combined with injection of the reactive Ar + 5%O₂ gas at a location 20 cm downstream of the atomization nozzle. This combination was intended to permit uninterrupted (low oxygen) atomization processing, but to provide reactive gas during freefall of the atomized particles at a location where they would still retain sufficient heat to promote excess surface oxidation. The exterior surfaces of the powder from the current atomization experiment (see Fig. 8b) appeared to be less rumpled, with more visible evidence of the underlying solidification structure of each atomized particle than the previous powders in Fig. 8a. Also, the current powders appeared to be more uniformly spherical, consistent with a less dominant surface oxide. The difference in surface appearance and shape of the atomized powders also was consistent with the measurements of bulk oxygen content, where the current powders had a bulk oxygen content of 800 ppmw (0.08 wt.%), compared to the previous powders with 2.0 wt.% oxygen⁸. Both of these measurements were for < 45 μm dia. powders.

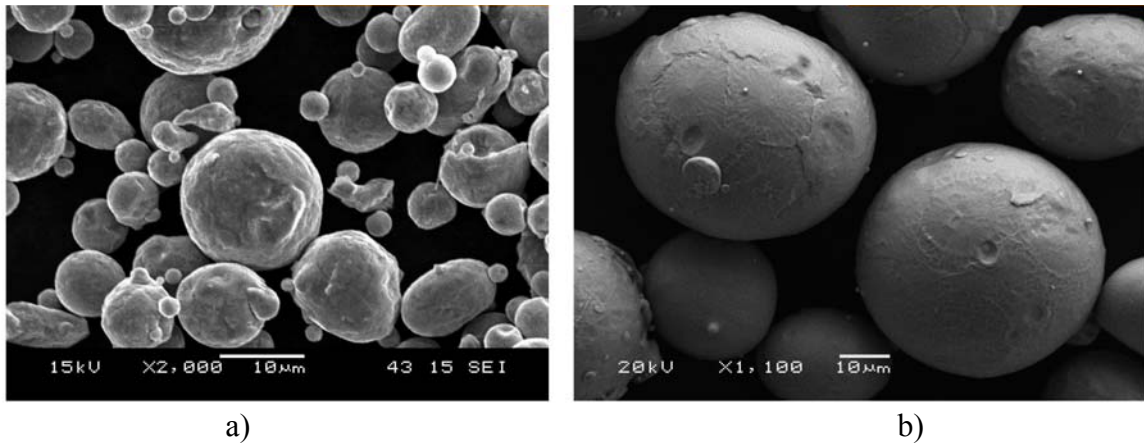


Figure 8. SEM micrographs of as-atomized Fe-12.5Cr-1Y powders that were produced with a) reactive atomization gas (Ar + 5%O₂), and b) inert (Ar) atomization gas with a downstream reactive gas (Ar + 5%O₂) injection.

The reactive gas atomized Fe-12.5Cr-1Y powder from the previous experiments was coated with a significant layer (1-3 µm thick) of Cr₂O₃ oxide, as demonstrated in cross-section micrographs (see Fig. 9). The SEM micrograph in Fig. 9 also illustrates that the oxide coating, or “shell,” had a tendency to spall from each particle during the powder handling involved with sieving (20-53 µm size fraction shown) and metallographic mounting. Cross-sections of the current Fe-12.5Cr-1Y powder did not reveal any apparent oxide coating, but surface analysis by Auger electron and X-ray photoelectron spectroscopy is planned to characterize the powder surfaces after downstream gas reaction processing.

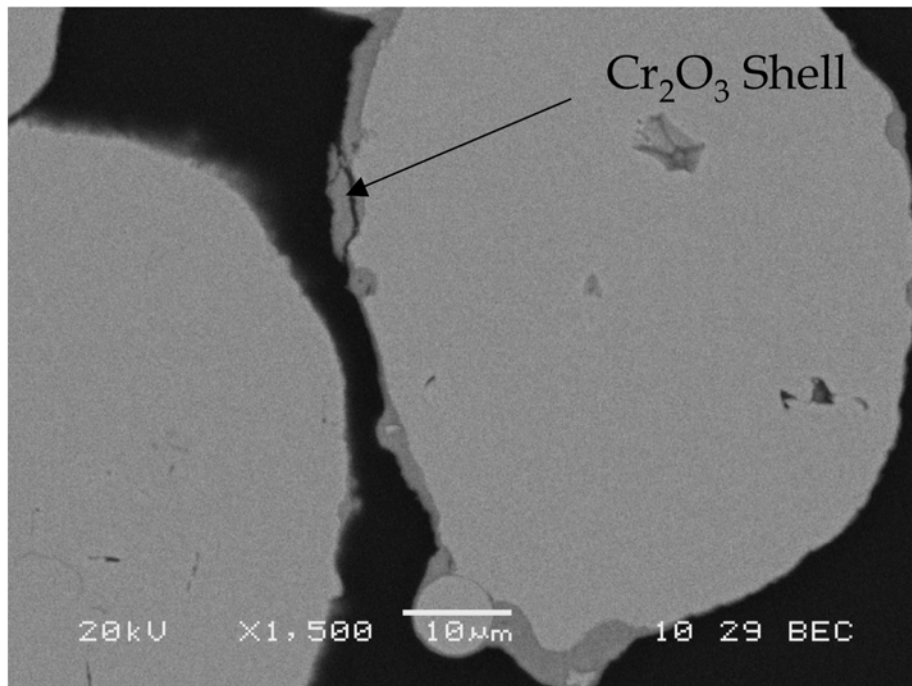


Figure 9. SEM cross-section micrograph of 20-53 µm powder from a previous reactive atomization experiment with evidence of spalling of the Cr oxide surface coating.

Further characterization of HIP consolidated Fe-12.5Cr-1Y powder from a previous reactive gas atomization experiment also focused on gathering quantitative evidence of the exchange reaction between the Cr_2O_3 trapped at the prior particle boundaries (ppb) and the Y content dissolved in the Fe-rich matrix phase. For the as-consolidated sample shown in Fig. 10a, the HIP conditions were 303 MPa at 1300C for 2h, which produced a fully dense microstructure that showed signs (spheroidization and discontinuous layers) of initial dissolution of the Cr_2O_3 phase at the ppb, presumably releasing oxygen that can transport and react with dissolved Y in the matrix. Quantitative metallographic analysis revealed 7.3%, of the dark Cr_2O_3 phase in the as-HIP condition, compared with 5.7% after a further vacuum annealing treatment of 1300C for 2h (see Fig. 10b), showing evidence for additional progress of the intended oxide exchange reaction.

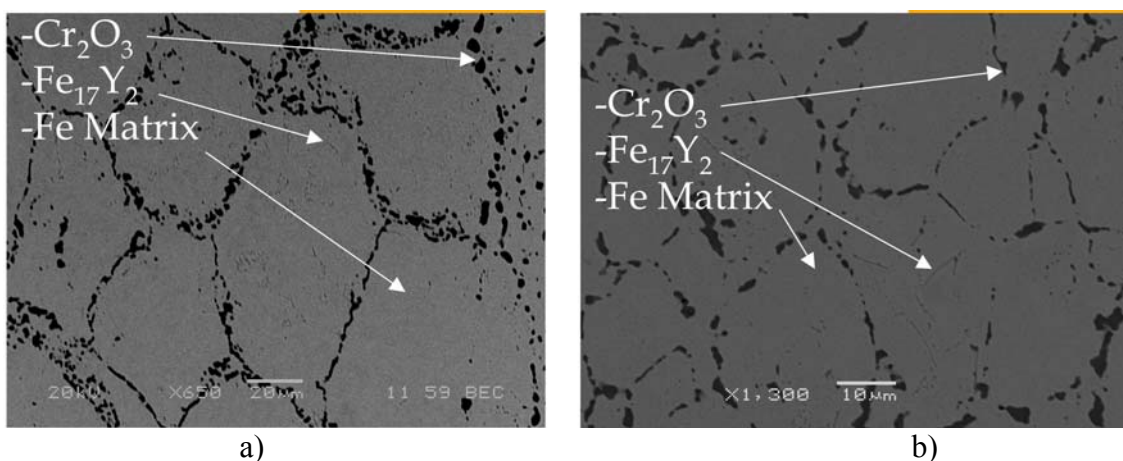


Figure 10. SEM micrographs in backscattered electron contrast of HIP consolidated microstructures made from reactive gas atomized Fe-12.5Cr-1Y powders from a previous experiment, showing, a) the as-HIP microstructure and, b) a consolidated sample after further annealing at 1300C for 2h.

New TEM results (see Fig. 11) were successful at EDS identification of numerous examples of Y oxide and Y-Cr-O dispersoids from 10-100 nm in diameter in the as-HIP microstructure made from Fe-12.5Cr-1Y powders of a previous reactive gas atomization experiment. The semi-quantitative identification of the dispersoid compositions and size agree with other observations of microstructures from similar ODS alloys (without Ti) made by mechanical alloying methods¹¹. While the intention of the alloy design was to generate a ferritic Fe-Cr matrix that could be heat treated in either austenitic or ferritic phase fields, preliminary results from high temperature X-ray diffraction analysis of < 20 μm dia. powder from a previous reactive gas atomization experiment showed some unexpected phase transformation behavior⁹. The first phase transformation occurs in the temperature range of 850-950C, where α -iron apparently transformed to an FCC iron oxide (FeO), termed wustite⁹. Upon further heating, a second transformation takes place in the temperature range of 1150-1200C, where FCC-FeO transforms to FCC γ -iron⁹. While these results are intriguing, the uncertain vacuum atmosphere in the high temperature (malfunctioning vacuum sensor) diffractometer makes it necessary to repeat and extend these measurements after repairs are completed. New bulk measurements of the crystal structure transformations as a function of temperature also are planned on the high intensity X-ray system of the Advance Photon Source at ANL to provide further verification. In addition, diffusion couple experiments have been initiated recently to investigate atomic transport kinetics and spatial distribution as a function of temperature for oxygen, Y, and Cr

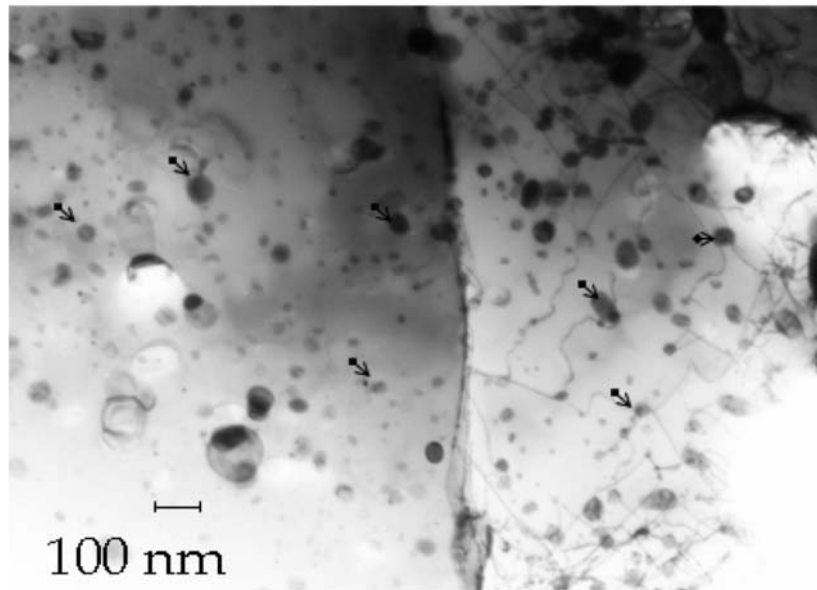


Figure 11. TEM bright field micrograph showing a significant population of spherical dispersoids in the as-HIP microstructure of a Fe-12.5Cr-1Y alloy, where small arrows mark the location of particles that were examined by EDS for approximate composition.

CONCLUSIONS

A new sample configuration and modified sintering conditions were successful at producing a near-optimum porous membrane support surface from $< 3\mu\text{m}$ spherical gas atomized Fe-16Al-2Cr (wt.%) powders. A chamfered edge was needed for the support tube needed to prevent rim cracking. The results indicated that to achieve an optimum membrane support surface, a secondary support frit with reduced pore size is needed to eliminate the remaining defects in the membrane support. Tests of the completed (with Pd thin film) hydrogen separation membrane system are needed to verify the effectiveness of an increased oxide diffusion barrier film at suppressing Fe and Cr diffusion into Pd. A novel processing technique for precursor powder involving gas atomization and in situ oxidation shows promise for simplified production of ODS ferritic stainless steel alloys with isotropic microstructure, compared to the current mechanical alloying approach that results in anisotropic mechanical properties. Atomization parameters have been demonstrated to control the concentration of oxygen (from 2 wt.% to 800 ppmw) introduced into the alloy system during in-situ oxidation of the precursor powder. Hot isostatic pressing of such powder was shown by SEM and TEM results to produce a desirable ODS microstructure. Preliminary heat treating experiments have proven the ability to manipulate the formation of the secondary strengthening dispersoid phase, suggesting that a heat treatment temperature of 1300C (similar to MA processing) is useful for oxide dispersion development. An effective heat treatment schedule is being formulated using high temperature in-situ x-ray diffraction and new diffusion couple experiments to control the final ODS microstructure. Mechanical property measurements also are planned for this ongoing work.

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