The Effect of Processing Parameters on Plasma Sprayed Beryllium for Fusion Applications

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ABSTRACT

Plasma spraying is being investigated as a potential coating technique for applying thin (0.1-5 mm) layers of beryllium on plasma facing surfaces of blanket modules in ITER and also as an in situ repair technique for repairing eroded beryllium surfaces in high heat flux divertor regions. High density spray deposits (>98% of theoretical density) of beryllium will be required in order to maximize the thermal conductivity of the beryllium coatings. A preliminary investigation was done to determine the effect of various processing parameters (particle size, particle morphology, secondary gas additions and reduced chamber pressure) on the as-deposited density of beryllium. The deposits were made using spherical beryllium feedstock powder which was produced by centrifugal atomization at Los Alamos National Laboratory (LANL). Improvements in the as-deposited densities and deposit efficiencies of the beryllium spray deposits will be discussed along with the corresponding thermal conductivity and outgassing behavior of these deposits.

1.0 Introduction

Fabrication and maintenance of surfaces that are directly exposed to the plasma in magnetic fusion energy (MFE) devices will present challenging problems in the development and design of the International Thermonuclear Experimental Reactor (ITER), the next generation magnetic fusion energy device. Plasma spraying technology is currently being evaluated as a potential method for fabrication and maintenance of plasma facing components (PFC’s) which will be subjected to severe environmental conditions as a result of either normal or off-normal operating conditions. At present, the layered armor materials for plasma interactive surfaces are beryllium, carbon, or tungsten [1,2].

Beryllium has been selected as the prime candidate for PFC’s for ITER due to some quite advantages over both carbon and tungsten [1,4].
One advantage of beryllium over carbon is the ability to use plasma spraying for in situ repair of damaged components in high heat flux regions of ITER i.e., divertors. Additionally, plasma spraying of beryllium is also being considered as a potentially large surface area deposition technique for coating first wall blanket modules made from either vanadium or stainless steel. Coating these modules with a thin (0.1-1mm) layer of beryllium will protect the underlying walls from the attack of oxygen or water and provide at the same time an acceptable plasma facing material.

Extensive investigations of plasma spraying of beryllium were done during the late 1960's and early 1970's by Union Carbide's Spectra-Lab and the Atomic Weapons Research Establishment (AWRE) for a number of defense related projects. Processing conditions and the thermophysical properties of plasma-sprayed beryllium produced during this period can be found in the book Beryllium Science and Technology, Volume 2, which was published in 1976 [8]. These investigations focused on the process optimization of beryllium plasma spraying using state of the art equipment available at that time. Deposit densities of beryllium were reported to be approximately 90% of beryllium's theoretical density (1.85 g/cm³) with porosity levels on the order of 10-13% in the as-sprayed condition. These levels are substantially less than densities required for ITER (95% dense material with 1-2% porosity). Lower porosity levels are needed in order to maximize the thermal conductivity of the spray deposit in order to transfer heat away from components in high heat flux regions of ITER.

A limited amount of research on the plasma spraying of beryllium for magnetic fusion energy devices has been reported in the recent literature [6,7]. The most recent investigations conducted by Battelle, Columbus in 1986, showed an improvement in the as-sprayed densities over results which were achieved by both AWRE and UCL. Results were reported to be on the order of 60 to 95% [9]. These levels were far short of early claims by Battelle of 95% dense plasma sprayed beryllium with 5% deposit efficiency (fraction of material deposited on a surface) [8]. In all of the previous investigations, plasma spraying of beryllium was done under a controlled inert environment at atmospheric pressure.

Advances in plasma spray technology over the past decade have shown that by operating under reduced pressure conditions some dramatic substantial increases in the as-deposited densities can be achieved [10]. This process technology was that introduced by Matsushita et al. [11] in 1970 and has been termed HIPPS (High Pressure Plasma Sprayer). A number of factors which contribute to the increased quality of plasma sprayed deposit are:

- Higher particle velocity, which result in better impacting and adhesion of the liquid particle,
- transferred arc heating/cleaning of the substrate which improves deposit density and adhesion,
- broad spray patterns for coating large surface areas,
- and the ability to spray reactive materials under a protective atmosphere.

A large number of processing variables can ultimately affect the quality of the beryllium spray deposits when spraying under a reduced pressure. Operating parameters which have a direct influence on the dwell time of the injected beryllium powders will significantly affect the degree of melting of the beryllium powder feedstock. Investigations have shown that powder size, powder morphology, powder injection, particle velocity, and the heat content (enthalpy) of the plasma, can substantially change the melting behavior of the injected powders [10].

In this investigation, results will be presented on deposits of beryllium produced by low pressure plasma spraying. The effects of powder size, powder morphology and the use of helium as a secondary plasma gas on the as-deposited density will be discussed. Characterization of the beryllium spray deposits will include optical microscopy, chemical analysis, thermal conductivity and outgassing studies.

2.0 Experimental Details

2.1 Beryllium powder production

Los Alamos National Laboratory has recently been investigating the centrifugal atomization process for producing spherical beryllium and beryllium alloy powders as feedstock material for advanced consolidation processes i.e., hot isostatic pressing (HIP) and plasma spraying. The centrifugal atomization process involves vacuum induction melting of a beryllium metal charge in a Mo crucible while directing the molten beryllium metal through a Zirc transfer tube onto the surface of a rapidly spinning wheel which is driven by an air turbine, Fig. 1. The liquid beryllium metal is mechanically atomized into finely divided droplets at the periphery of the spinning wheel. The droplets are solidified in flight inside the atomizing chamber by a transverse flow of helium gas that also carries the beryllium powder product into a cyclone separator. This process produces spherical beryllium powder with solidification rates on the order of 10^4 K/s.
degrees C/sec. With the current equipment configuration, the beryllium powder is collected in a canister below the cyclone separator, valved off, and transferred to an inert-gas dry box for powder classification. Parameters which can influence the resulting beryllium powder size distribution are given in Table 1.

Table 1. 
Parameters which can influence the size distribution of centrifugally atomized beryllium powders.

<table>
<thead>
<tr>
<th>Melt temperature</th>
<th>Pour temperature</th>
<th>Superheat</th>
<th>Disk speed</th>
<th>Nozzle diameter</th>
<th>Atmosphere</th>
</tr>
</thead>
</table>

The powders produced by the centrifugal atomization process were screened using stainless steel sieves to the following size fractions.

A. 400 mesh (< 45 μm)
B. 270-400 mesh (53 μm - 75 μm)
C. 200-270 mesh (75 μm - 90 μm)
D. 140-200 mesh (75 μm - 106 μm)

Particle size fractions A, B, C, D were selected for this investigation because they contained the largest fractional yields of the atomized powders and also represented a wide range of particle sizes.

2.2 Beryllium plasma spraying

Spray deposits of beryllium were made using the low pressure plasma spray chamber at LANL, Fig. 2. This chamber contains a commercial Scintrex PlasmaWoman torch which is mounted over a translating copper cooled substrate. Accurate control of the processing gases used in the plasma spray process was accomplished using a multi gas flow control system. Beryllium powder was fed into the plasma spray torch by using a weight feed control system which integrates a commercial powder feeder with a Telos weight scale to measure and control the feed rate of the beryllium powder feedstock during the spray.
operation. Plasma-spraying of beryllium using this facility can be done under either a reduced pressure environment or in an argon atmosphere.

To understand the effect of processing parameters on the as-deposited densities and deposit efficiency of plasma-sprayed beryllium, parameters were changed from an initial standard operating condition which was established using commercial S-65 beryllium powder from Brush Wellman Inc., Table 2.

**Table 2. Standard Operating Condition**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spray torch</td>
<td>Plasmadyne SG-100</td>
</tr>
<tr>
<td>Current</td>
<td>700 amps</td>
</tr>
<tr>
<td>Voltage</td>
<td>30 volts</td>
</tr>
<tr>
<td>Primary arc gas (Ar)</td>
<td>30 slm</td>
</tr>
<tr>
<td>Powder carrier gas (Ar)</td>
<td>2.5 slm</td>
</tr>
<tr>
<td>Powder feed rate</td>
<td>.5 lbs/hr</td>
</tr>
<tr>
<td>Spray distance</td>
<td>76.2 mm</td>
</tr>
<tr>
<td>Substrate translation</td>
<td>39 ipm</td>
</tr>
<tr>
<td>Atmosphere</td>
<td>argon</td>
</tr>
<tr>
<td>Anode/Cathode</td>
<td>145/290</td>
</tr>
</tbody>
</table>

The following processing parameters were investigated:

- particle size and particle morphology
- secondary gas addition, (helium)
- chamber pressure

Spray deposits of beryllium were made on (4.175mm) thick copper substrates which were translated back and forth under the plasma spray torch. A bell shaped deposit was produced on the copper substrates after spraying for 5 minutes. Deposit thicknesses ranged from 2mm to 10mm at the thickest region of the deposit with deposit lengths on the order of 50mm. Removal of the beryllium deposits from the copper substrates for subsequent characterization was done by bending the substrate until the deposits detached. As deposited densities of the beryllium spray deposits were measured using a water immersion technique (Archimedes principle). Measurements were made on the thickest region of the spray deposits after cutting/polishing to remove the outer fringes of the bell shaped spray deposits.
Deposit efficiencies (the fraction of material deposited on the substrate) were determined by measuring the weight of the substrate before and after depositing beryllium. The difference in weight was compared to the total amount of beryllium powder dispensed during the plasma spray run. Since the beryllium powder feed-rate is controlled by a weight/loss system which places the powder feed hopper on a weight scale, an accurate amount of powder present in the hopper before and after the spray operation could be determined.

2.3. Deposit Characterization

2.3.1 Microstructural and Chemical Analysis

Characterization of the beryllium spray deposits in the as-deposited condition was accomplished using polarized light microscopy and chemical analysis. Spray deposits were cross-sectioned, mounted and polished for viewing in polarized light. Samples were etched with a solution containing 3% HF, 3% HNO₃, and 3% H₂SO₄ for 3 to 5 seconds to determine the microstructure of the as-sprayed deposits. Chemical analysis was performed by Brush Wellman Inc., Elmore Ohio, on the centrifugal atomized beryllium powders, the beryllium plasma spray deposits and the beryllium ever-sprayed powder using a combustion analysis technique.

2.3.2 Thermal Conductivity Measurements

The room temperature thermal diffusivity was measured on free standing beryllium samples 6mm in diameter by 5mm long. Measurements were made at Oak Ridge National laboratory using a thermal pulse technique in which one face of the specimen was illuminated with a xenon flash lamp and the other was monitored with an IR detector. The temperature as a function of time is plotted and the thermal diffusivity is calculated from the resultant experimentally determined thermal transient. The thermal diffusivity (D) was calculated using the following expression:

\[
D = \frac{1.17 \alpha}{\pi h},
\]

where \( h \) is the specimen thickness and \( t \) is the time required for the back face of the specimen to reach half its maximum temperature. The thermal conductivity (\( \kappa \)) of the beryllium spray deposits was calculated using the following expression:

\[
\kappa = \frac{pC_p}{h}.
\]
where $\rho$ is the density and $C_p$ is the heat capacity.

2.3.3 Vacuum Outgassing

Thermal desorption spectra were obtained on several samples of plasma-sprayed beryllium at Sandia National Laboratory, Livermore, California, to determine their vacuum outgassing characteristics. The outgassing system consisted of a turbo-pumped, ultra-high vacuum quartz tube furnace with a UTL 100°C residual gas analyzer (RGA). The measurements were made on small block-shaped samples (100mg, 5x5x2 mm) by first loading them into an unheated portion of the furnace and pre-outgassing the tube to 800°C to remove the contaminants resulting from air exposure. The samples were then remotely transferred into the heated zone for thermal desorption. Several samples were run at the same time to gain sufficient signal for accurate measurements. The sample temperature was ramped linearly from room temperature to 600°C at 10 °C/min using a temperature programmer. For one experimental run (#2), the temperature ramp was paused at 350°C for 90 minutes to examine the effect of vacuum baking at 350°C. RGA signal amplitudes were converted to gas partial pressures by calibrating against carbon monoxide and hydrogen standard leaks.

3.0 Results and Discussion

3.1 Beryllium deposit density and microstructure

The effect of processing parameters (particle size, helium gas addition and low pressure plasma spraying) on the as-deposited densities are given in Table 3.

Table 3

<table>
<thead>
<tr>
<th>Particle size (mesh)</th>
<th>Standard spray condition</th>
<th>Secondary gas (He) addition</th>
<th>Reduced pressure (40 torr)</th>
<th>Secondary gas reduced pressure</th>
</tr>
</thead>
<tbody>
<tr>
<td>400 (44 μm)</td>
<td>88.44%</td>
<td>92.54%</td>
<td>94.14%</td>
<td>94.49%</td>
</tr>
<tr>
<td>270 (40 μm)</td>
<td>91.8%</td>
<td>92.5%</td>
<td>94.2%</td>
<td>94.1%</td>
</tr>
<tr>
<td>140 (48 μm)</td>
<td>90.8%</td>
<td>90.0%</td>
<td>90.0%</td>
<td>90.0%</td>
</tr>
<tr>
<td>100 (75 μm)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The highest deposit densities were achieved using the beryllium atomized powders which were below 38\textmu m. The spherical morphology of the atomized powders, Fig. 3a, allowed for better feeding of powders into the spray torch than commercially impact ground beryllium powders which are more angular and difficult to feed below 45\textmu m Fig. 3b. Beryllium atomized powders in the size range of 53-38\textmu m also showed relatively good deposit densities (91-92\% T.D.) when spraying with the helium gas addition at a reduced pressure of 350 torr. The larger beryllium feedstock powders (106-45\textmu m) were difficult to melt using the standard operating conditions and required an increase in the operating current from 700 amps to 800 amps in order to melt the beryllium and adhere to the copper substrates. The deposit densities (<60\%) were considerably lower and did not significantly change with the introduction of the helium secondary gas and the low pressure spray environment. No further analysis was done on these spray deposits which were made from large diameter powders.

A graph of the as-deported density, deposit efficiency and the level of porosity of plasma-sprayed 38\textmu m beryllium powder under the various processing conditions is given in Fig. 4. The highest deposit density (94.9\%) was achieved using the standard operating parameters given in, Table 2, with the addition of 15 standard liters per minute (SLM) of helium as a secondary plasma gas while operating under a reduced pressure (350 torr). This operating condition also resulted in the highest deposit efficiency (approximately 60\%) with a porosity level on the order of 4 percent. These observed increases were attributed to the higher heat content (enthalpy) of the plasma jet with the addition of the helium secondary gas, and the higher particle velocities that result when spraying under a reduced pressure. Increased particle velocities improve the impacting and splatting of the melted and partially melted beryllium feedstock powders resulting in better bonding and consolidation of the deposit.

Microstructural analysis of low density beryllium plasma sprayed deposits with density levels on the order of 90\%, show a large population of unmelted beryllium particles with corresponding porosity adjacent to these unmelts, Fig. 5a. In the case of the higher density beryllium deposits, the presence of unmelted beryllium particles decreased with a corresponding decrease in porosity level, Fig. 5b. Unmelted beryllium particles were still present in the deposits although a greater degree of consolidation of the unmelts seemed to occur during low pressure plasma spraying.
3.2 Chemical Analysis

Analysis of the -400 mesh (38μm) beryllium atomized powders, beryllium spray deposits and the over-sprayed beryllium powder (which was collected on the bottom of the spray chamber) was performed to determine the oxygen level and other impurity elements. The oxygen levels of these samples were compared to commercial SP-65 beryllium powder produced by Brush Wellman, Inc., and beryllium plasma-sprayed deposits produced by Battelle, Columbus, Fig. 6.

The oxygen level of the plasma sprayed beryllium deposits (.35%) was approximately half that of the starting atomized beryllium powder (.65%) and much lower than the over-sprayed beryllium powder (1.15%). In addition, the oxygen content of the spray deposits was lower than what was previously reported in earlier investigations done by the UCSL and AWRE [11]. Oxygen levels in the beryllium spray deposits in these investigations were reported to have increased from approximately 20 to 140 percent over the starting beryllium feedstock powder.

Metallic powders which were deposited using low pressure plasma spraying have shown oxygen levels in the spray deposits at least as high as the levels present in the starting feedstock powders. When plasma-spraying copper using hydrogen as a secondary plasma gas, oxygen levels in the spray deposits were shown to have decreased below the starting powders [7]. In this investigation, helium was used as the secondary plasma gas and should not have affected the oxygen level in the spray deposits.

The lower oxygen level in the beryllium spray deposits is not well understood but may be attributed to the plasma/particle interactions that occur when spraying beryllium powders which contain a surface layer of BeO. During melting, the powders may tend to segregate in beryllium and beryllium oxide particles due to their differences in melting points and densities (BeO-1.03 g/cm³, M.P.-2823K and Be-1.86 g/cm³, M.P.-1560K). This segregation may cause different particle trajectories of the Be and BeO particles as they exit the plasma torch. The BeO particles may volatilize in flight before impacting the substrate and subsequently deposit off the substrate. Additionally, the BeO particles may be significantly smaller than the Be particles and become entrained in the processing gases which are deflected around the substrate. These two possibilities may account for the lower oxygen levels present in the over-sprayed powders. Further investigations are being done to understand these results.

Elevated levels of Fe, Ni, Cu were also detected in the atomized beryllium powders, and the subsequent sprayed deposits when compared to commercial impact ground SP-65 beryllium powder manufactured by
Brush Wellman, Inc. These elevated levels are a result of the erosion that occurs in the inner walls of the stainless steel cyclone separator during the beryllium powder production. Efforts are underway to coat the inside surfaces of the cyclone separator with plasma sprayed beryllium in order to minimize the contamination of the beryllium powders through impact with the stainless steel walls.

3.3 Vacuum outgassing of plasma-sprayed beryllium

The observed quantities of outgassed species per gram of sample material are given in Fig. 7. The major gas species are water vapor (18 amu) and hydrogen (2 amu). Measurable quantities of methane (16 amu), carbon monoxide or nitrogen (28 amu), and ammonia (17 amu) are also observed. Here, the NH₃ and CH₄ values are the residual amplitudes for 17 and 16 amu obtained after subtracting off the fragmentation contributions from H₂O and NH₃, respectively. Neither the interruption of the temperature ramp performed in experiment (2) nor the size difference of the two sample sets affected the total quantities released per gram. The hydrogen released is roughly the same for the two experimental runs; however, more was released in the forms of NH₃ and CH₄ in run #1. Typical quantities outgassed from 85% and 95% dense, S-65 beryllium samples from Brush Wellman, Inc., are also given for comparison.

Much of the outgassing behavior of the plasma-sprayed samples is dominated by the presence of the large H₂O signal. It is not known whether this signal is typical of plasma sprayed material (resulting from post-processing exposure to air) or is an artifact of the specific sample pretreatment. Prior to outgassing, porosity and density measurements were done on these samples by immersing each in water. The H₂O probably resides on the oxide present on the surface of the spray deposits. BeO is known to be very hygroscopic and accommodates several waters of hydration, forming BeO·xH₂O.

The thermal desorption spectra for the plasma sprayed (PS) material is shown in Fig. 8. The water is weakly bound and can be desorbed at low temperatures. Hydrogen production at higher temperatures probably results from the reaction of residual H₂O with beryllium at these temperatures. Desorption spectra for the other species are compared with S-65 spectra in Fig. 9. The PS material exhibits a low temperature N₂/CO peak (28 amu) not found, or weakly present, in S-65. Most of the NH₃ detected for the PS material occurs at this lower temperature. The CH₄ increase for the lower porosity materials also occurs at a lower temperature.

Pre-baking the PS material at 450 °C for 30 minutes removes the water peak, but has little effect on the high temperature hydrogen peak. It also removes the low temperature N₂/CO peak without affecting the high temperature N₂/CO peak, as shown in Fig. 10. From careful analysis of companion mass peaks, particularly 12, 14, and 16 amu, it can be
concluded that this low temperature peak is N\textsubscript{2}, whereas the high temperature 28 amu peak is CO. Apparently, it is the presence of weakly bound N\textsubscript{2} which gives rise to the formation of NH\textsubscript{3}. This nitrogen may result from post-process absorption from air or may be due to nitrogen present in the initial powder feedstock. It also may be present in the processing gases as was described in reference [11]. If present in the powder, nitrogen may be removed by outgassing the beryllium powder feedstock prior to spraying. Other weakly-bound contaminants, including water, should also be removable by a pre-outgassing step. As mentioned above, removing the water may also reduce much of the detected hydrogen. However, experiments investigating air exposure of outgassed S-65 have shown both N\textsubscript{2} and H\textsubscript{2} rapidly return to near their pre-outgassed levels. Thus air exposure must be prevented following such a pre-outgassing.

Water outgassing from the PS material continued throughout each experiment, but varied somewhat with sample temperature, T. Even after baking at 600 °C for 90 minutes, substantial H\textsubscript{2}O outgassing remained. Figure 11, gives the H\textsubscript{2}O outgassing as a function of time for the PS and 85% dense S-65 materials. Each data set can be fit with an exp(-t\textsuperscript{1/2}) function indicating diffusion-controlled release. Thus, although the water is weakly bound, probably in the oxide on the surface of each deposit, it appears to follow a very tortuous path to release. Outgassing from the PS material is substantially slower and will require a much longer pumpdown time.

### 3.4 Thermal Conductivity

Results of the room temperature thermal conductivity measurements are given in Table 4.

**Table 4.**

Room temperature thermal conductivity of plasma-sprayed beryllium

<table>
<thead>
<tr>
<th>Sample</th>
<th>Mean Thermal Diffusivity (cm\textsuperscript{2}/s)</th>
<th>Specific Heat (J/kg·K)</th>
<th>Density (g/cm\textsuperscript{3})</th>
<th>Thermal Conductivity (W/m·K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.2269</td>
<td>1750</td>
<td>1.7500</td>
<td>69.44</td>
</tr>
<tr>
<td>B</td>
<td>0.2145</td>
<td>1750</td>
<td>1.7141</td>
<td>64.34</td>
</tr>
<tr>
<td>C</td>
<td>0.1586</td>
<td>1750</td>
<td>1.6702</td>
<td>46.36</td>
</tr>
<tr>
<td>D</td>
<td>0.1442</td>
<td>1750</td>
<td>1.6653</td>
<td>42.02</td>
</tr>
<tr>
<td>E</td>
<td>0.1567</td>
<td>1750</td>
<td>1.6668</td>
<td>45.71</td>
</tr>
<tr>
<td>F</td>
<td>0.1246</td>
<td>1750</td>
<td>1.6997</td>
<td>37.06</td>
</tr>
</tbody>
</table>
The thermal conductivity of the beryllium spray deposits were significantly lower than that of pure beryllium which has a thermal conductivity of 218 W/m·K [12]. These results were in general agreement with previous thermal conductivity data taken for plasma sprayed beryllium produce by inert gas plasma-spraying at Battelle, Columbus [8]. An increase in the thermal conductivity also corresponded to an increase in the deposit density, except for sample F. The low thermal conductivity values in all cases can be attributed to the porosity and layered microstructure throughout the bulk of the deposit, Fig.5. The presence of interfaces created by impacting beryllium liquid particles will be a controlling factor in maximizing the thermal conductivity of the spray deposits. Improvements in the thermal conductivity of plasma sprayed beryllium can result by minimizing the splat interfaces through better melting and deposition, and also through post heat-treatments. Spray deposits of beryllium which were produced by Battelle, Columbus were heat-treated at 900°C for 1 hour to promote diffusion bonding across splat interfaces. An increase in the thermal conductivity from 25-200% over the as-sprayed beryllium resulted [8]. Since heat-treatments or consolidation by hot isostatic pressing will not be applicable for ITER, post-deposition surface conditioning techniques such as laser surface treatments should be investigated.

4.0 Conclusions

- The spherical nature of the beryllium centrifugal atomized powders, in comparison to the angular impact ground powders, allowed for better feeding of powders below 38μm into the plasma-spray torch.

- 38μm spherical beryllium powder, made by centrifugal atomization, produced the highest deposit densities under the investigated conditions.

- Increases in the deposit density and deposit efficiency of plasma sprayed beryllium resulted when spraying under a low pressure condition (350 torr) with helium as a secondary plasma gas.

- Oxygen levels in the beryllium spray deposits produced by low pressure plasma spraying were lower (by a factor of two) than the starting atomized beryllium powders.

Outgassing of plasma sprayed beryllium was dominated by the presence of H2O and H2.

The thermal conductivity of plasma sprayed beryllium was significantly lower than pure beryllium. Microstructural features such as splat interfaces may be a controlling factor.
Acknowledgments

This investigation was funded through Sandia National Laboratory, Fusion Technology Department under the guidance of Dr. Robert D. Watson. The authors would like to acknowledge Oak Ridge National Laboratory for the thermal conductivity measurements, K. Elliott for the beryllium plasma-spraying and J. Montoya for sample preparation and metallography.
References


8. Private communication with Dr. R.D. Watson of Sandia National Laboratory, Fusion Technology Division, Albuquerque, N.M. USA.


Fig. 1. Schematic of centrifugal atomization process.
Fig. 2. Beryllium low pressure plasma spray facility at LANL.
Fig. 4. As-deposited density, deposit efficiency and porosity level of plasma sprayed (<50µm) beryllium powder sprayed under the various operating conditions and compared to results previously reported by Battelle, Columbus [8].
Fig. 6. Comparison of oxygen levels of commercial SP-65 beryllium powder from Brush Wellman Inc., plasma sprayed beryllium deposits produced at Battelle, Columbus, centrifugal atomized beryllium powders produced at LANL, beryllium spray deposits produced at LANL, and the over-sprayed powders.
Fig. 7. Quantities of gaseous produced by heating as received plasma sprayed (PS) and sintered materials of the indicated densities.
Fig. 8. Gas desorption spectra from 94% dense plasma sprayed beryllium samples.
Fig. 9. Comparison of the N$_2$/CO, NH$_3$, and CH$_4$ desorption spectra for plasma sprayed (PS) and S65 material of the indicated densities.
Fig. 18. Comparison of desorption spectra for (PS) material pre-baked at 950°C for 30 minutes with unbaked material. Pre-baking removed the water peak but not the water on-grafting.
Fig. 11. Water release rate at 600°C.
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Workshop on Beryllium for Fusion Applications
Karlsruhe, Germany
October 4-5, 1993
Outline

- Facilities
- Applications
- Requirements
- Preliminary Results
- Future Activities
Centrifugal Atomizer Schematic

- Stopper rod
- Crucible
- Molten alloy
- Induction coil
- Melting chamber
- Transfer tube
- Helium quench gas
- Nozzle
- Alloy stream
- Atomized droplets
- Powder exit
- Atomizer wheel
- Air turbine
XSR Powder Size Distributions

- Size (μm): <44, 44-62, 62-105, 105-125, 125-177, >177
- Weight Percent of Powder

Legend:
- Run 9
- Run 10
- Run 11
- Run 12
Plasma Spraying of Beryllium for ITER

● Applications:
  - In-situ repair of sputter eroded and disruption damaged beryllium armour tiles in high heat flux regions.
  - Fabrication of large area (1000 m²) beryllium coatings (1-2mm) over stainless steel or vanadium first wall surfaces.

● Requirements:
  - high density
  - high deposit efficiency
  - good thermal conductivity
  - good bond strength between coatings and substrate materials (Be, S.S etc.)
  - enhanced mechanical behavior under pulse fusion conditions
  - others......
High Energy Systems

Divertor Target Concepts

Water

Helium

Liquid Metals

Armored Circular Tube

Monoblock

Armored Rectangular Tube

Slitted Duct

McDonnell Douglas Aerospace
SATELITE Plasma Sprayed BERYLLIUM
Optimizing Spray Deposits of Beryllium

- Increase particle melting:
  - particle morphology
  - particle size distribution
  - substrate temperature
  - particle dwell time:
    - low velocity laminar flow
    - high flame temperature

[Image: Micrograph showing splat layers, unmelted particles, and porosity. 200X magnification.]
Layered Assembly of Impacting Discs

H. Herman, 1988
Operating Parameters for Plasma Spraying Beryllium

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary gas</td>
<td>(Ar) - slpm</td>
<td>30.0</td>
</tr>
<tr>
<td>Powder gas</td>
<td>(Ar) - slpm</td>
<td>2.5</td>
</tr>
<tr>
<td>Powder feed rate</td>
<td>grams/min</td>
<td>3.8</td>
</tr>
<tr>
<td>Spray distance</td>
<td>cm</td>
<td>7.6</td>
</tr>
<tr>
<td>Translation speed</td>
<td>cm/min</td>
<td>99.0</td>
</tr>
<tr>
<td>Chamber pressure</td>
<td>torr</td>
<td>500.0</td>
</tr>
<tr>
<td>Oxygen level</td>
<td>ppm</td>
<td>100.0</td>
</tr>
<tr>
<td>Current</td>
<td>amps</td>
<td>700.0</td>
</tr>
<tr>
<td>Leak-up rate</td>
<td>millitorr/min</td>
<td>5.0</td>
</tr>
<tr>
<td>Substrate (Cu)</td>
<td>mm</td>
<td>3.2</td>
</tr>
<tr>
<td>No. of passes</td>
<td></td>
<td>140.0</td>
</tr>
<tr>
<td>Spray time</td>
<td>min</td>
<td>5.0</td>
</tr>
<tr>
<td>Total powder sprayed</td>
<td>grams</td>
<td>9.0</td>
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</tbody>
</table>
Deposit Density and Deposit Efficiency of Plasma Sprayed Beryllium Under Various Conditions
- 400 mesh centrifugal atomized powders

Previous investigations
(Battelle)
A Comparison of Oxygen Levels in Beryllium Spray Deposits and Beryllium Powders

<table>
<thead>
<tr>
<th>Sample</th>
<th>Wt% Oxygen</th>
</tr>
</thead>
<tbody>
<tr>
<td>SP-65 deposits</td>
<td>0.6</td>
</tr>
<tr>
<td>Battelle deposits</td>
<td>0.84</td>
</tr>
<tr>
<td>Atomized powders</td>
<td>0.65</td>
</tr>
<tr>
<td>LANL deposits</td>
<td>0.35</td>
</tr>
<tr>
<td>Over-sprayed powders</td>
<td>1.15</td>
</tr>
</tbody>
</table>
Outgassing Behavior of Plasma Sprayed and S-65 Beryllium

![Graph showing outgassing behavior of different materials.]
Water Release Rate at 600 °C for Plasma Sprayed and S-65 Beryllium

Water Release at 600°C

93% dense PS
exp [ -(t/0.8 hr)]

85% dense S-65
exp [ -(t/0.3 hr)]
Plasma Spraying of Beryllium for ITER

Critical Research Areas:

- Optimize plasma spray parameters to produce high density/high thermal conductivity deposits of beryllium.

- Optimize centrifugal atomization process to produce high yields of low oxide, -400 mesh spherical beryllium powder.

- Investigate surface preparation techniques on the bond strength of plasma-sprayed beryllium.

- Evaluate the performance of plasma-sprayed beryllium coatings under pulse fusion conditions.

- Fabricate a robotically controlled plasma-spray test cell to evaluate remote manipulation and in-situ repair.