Studies on the preparation and plasma spherodization of yttrium aluminosilicate glass microspheres for their potential application in liver brachytherapy

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Abstract. Plasma spheroidization exploits the high temperature and high enthalpy available in the thermal plasma jet to melt irregularly shaped powder particles and quench them to get dense spherical particles. Plasma spheroidization is a versatile process and can be applied to metals, ceramics, alloys and composites to obtain fine spherical powders. Radioactive microspheres incorporated with high energetic beta emitting radioisotopes have been reported to be useful in the palliative treatment of liver cancer. These powders are to be prepared in closer range of near spherical morphology in the size range 20-35 microns. Inactive glass samples were prepared by heating the pre-calculated amount of glass forming ingredients in a recrystallized alumina crucible. The glass was formed by keeping the glass forming ingredients at 1700ºC for a period of three hours to form a homogeneous melt. After cooling, the glass was recovered from the crucible by crushing and was subsequently powdered mechanically with the help of mortar and pestle. This powder was used as the feed stock for plasma spheroidization using an indigenously developed 40 kW plasma spray system. Experiments were carried out at various operating parameters. The operating parameters were optimised to get spheroidised particles. The powder was sieved to get the required size range before irradiation.

1. Introduction

Radioactive microspheres incorporated with high energetic beta emitting radioisotopes have been reported to be useful in the palliative treatment of liver cancer [1]. In such an application, tiny radioactive microspheres, in the particle size 20-35 microns are injected into the hepatic artery of the patient, where they get trapped inside the blood vessels in the tumor and block the nutritional supply to the tumor in addition to providing a large and localized radiation dose by highly ionizing beta particles. $^{89}$Y is a naturally occurring isotope that can be incorporated in glass matrices and when irradiated with a beam of neutrons, captures the neutron to yield $^{90}$Y that is a beta emitter, emitting highly energetic $\beta$-rays $E_{\text{max}}$ of ~2.28 MeV and half life of 64 hours. In order to prepare a glass that on subsequent neutron irradiation produces radioactive glass, a composition containing 17Y$_2$O$_3$-19Al$_2$O$_3$-64SiO$_2$ (mol %) was chosen for the present work [2]. Spheroidization of aluminosilicate glass powder was done using a 40 kW DC plasma spray system. A 40 kW DC non-transferred arc plasma torch was used as the plasma source to conduct the experiments.
2. Sample preparation
Inactive glass samples were prepared by heating the precalculated amount of glass forming ingredients at 1700°C for a period of three hours to form a homogeneous melt. After cooling the glass was recovered from the crucible after cooling, and powdered by mechanical crushing. The powder obtained was sieved using standard test sieves to get 20-35 micron size powder for plasma spheroidization experiments. Particle morphology of the feed stock powder is shown in figure 1.

Spheroidization of aluminosilicate glass powder was done using a 40 kW DC plasma spray system. A 25 kW DC non-transferred arc plasma torch based system was used as the plasma source to conduct the experiments.

Figure 1. Feed stock aluminosilicate glass powder before spheroidization

3. Plasma Spheroidization

3.1 Experimental set-up
An indigenously built 40 kW atmospheric plasma spray system was used for spheroidization of aluminosilicate glass powder. This system consists of a 40 kW DC non-transferred arc plasma torch, Power supply, control console, powder feeder and a cooling water circulator. Schematic diagram of the system is given in figure 2.

3.2 Experimental procedure
Aluminosilicate glass powder was injected into the plasma jet by means of a volumetric turn table powder feeder. The powder passing out of the plasma flame was quenched and collected below in a stainless steel vessel at a distance of about 250 mm. The particles enter the plasma jet melt in the flame and get spheroidized due to surface tension forces. The estimated quench rate of the particle is of the order of $10^5$ - $10^6$ K/Sec. Spheroidization was carried out at different plasma power ranging from 8 kW to 20 kW. It is found that the plasma power strongly influences the degree of spheroidization. Experiments were carried out at various operating parameters to study the effect of spheroidization of the powder. Aluminosilicate powder of particle size 20-35 micron size prepared by crushing and sieving was fed into the plasma flame through a 2mm dia. orifice at the exit of the plasma flame. The powder entering in the plasma flame got melted and was subsequently quenched to get spherical particles. Typical operating parameters are given in table 1.
Figure 2. Schematic of Plasma Spheroidization system

1. Plasma torch  2. Power Supply  3. Powder feeder  4. H.F.Ignitor  5. Control Console
6. Gas supply  7. Cooling water circulator  8. Exhaust  9. Powder collection

4. Results and Discussion

Degree of spheroidization of the powder in the plasma jet depends on the plasma melting, which in turn is decided by the plasma power and dwell time of the particles. Particle temperature depends on the plasma temperature and the plasma-particle heat transfer mechanism. Experimental parameters affecting these, in turn, are input power to the plasma torch, enthalpy of the plasma, composition and flow rate of the plasma gas, electro-thermal and efficiency of the torch [3]. These operating parameters are optimized by solving heat transfer and momentum transfer equations for particles of different sizes.

Particle morphologies of plasma-processed aluminosilicate glass powder is shown in Figure 3. The particles are seen to possess spherical or near-spherical morphology. X-ray diffraction pattern of the powder remained amorphous after plasma processing. Figure 4 shoes the X-ray diffraction pattern of the glass powder before and after plasma spheroidization.

Figure 3. Plasma Spheroidised aluminosilicate glass powder
Table 1. Typical operating parameters for plasma spheroidization

| Powder Input (kW) | 20 |
| Primary Plasma Gas flow rate, Argon (LPM) | 30 |
| Secondary Plasma Gas flow rate, Nitrogen (LPM) | 3 |
| Powder particle size (microns) | 20-35 |
| Powder feed rate (g/min) | 15 |
| Powder Carrier gas, Argon (LPM) | 12 |
| Torch to base distance (mm) | 250 |

5. Conclusions

Inactive yttrium aluminosilicate glass could be prepared in reproducible manner and plasma spheroidization of aluminosilicate glass powder was successfully carried out using atmospheric plasma spray system. Glass prepared by melting of precalculated amount of glass forming ingredients, was used as the starting material for spheroidization. Solidification, crushing and sieving of glass powder in required size of 20-35 micron size could be controlled and plasma parameters were optimized for spheroidization of glass powder. XRD studies indicate that, spherical particles of required size are obtained by plasma spheroidization without crystallization or alloy formation.
References

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