

# CONTROL OF SILICON PARTICLES VELOCITIES IN RF THERMAL PLASMA BY L.D.A. FOR PURIFICATION PROCESS

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## ABSTRACT

We have developed a fluidized bed system powder feeder. Particle velocity into the plasma jet has been picked up with a LDA technology. The first results show that the particles go out the injector at 5 m/s and reach the maximum velocity in the axial flow (21 m/s). The mean residence time of the particles into the plasma is 15 ms, which correspond to the necessary time for a total fusion according to a model developed in the laboratory.

## I INTRODUCTION

Processes involving the treatment of a powder by means of a thermal plasma have been widely studied. In order to avoid any pollution during the plasma treatment, it's necessary to control the chemical composition of the plasma around the particle. The residence time of particles in the plasma define the melting and evaporating mechanisms. To control the continuous feeding of a melted silicon bath or a spraying of thin film on a substrate, we developed a fluidized bed feeder system. It feeds axially the plasma jet with silicon particles in a large range by using argon carrier gas. The size of the particles injected in the plasma can be selected during the fluidized mechanism by different parameters. The RF plasma, operating at 25 kW, uses an axial and annular flow rate respectively equal to 30 l/min and 40 l/min (fig.1). The measurements are obtained by means of LDA system with 300 mW argon-laser source.

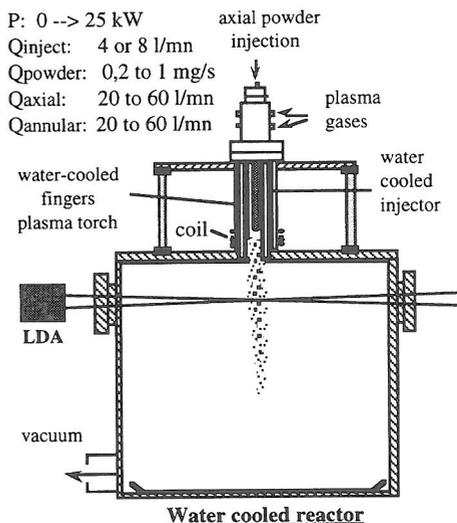


Fig.1

## II EXPERIMENTAL SET UP FOR A POWDER FEEDER

### 1 The powder feeder technology

The study of the powder behaviour in a thermal plasma jet requires a well adapted powder feeder. Its characteristics are described below (fig.2):

- the fluidized bed system permits on the one hand to control the gas composition and flow, on the other hand to avoid atmosphere pollution,
- powder movement is stayed by a vibrating generator system which permits to obtain a constant regular gas and particle flow,
- the powder collector can be fixed at different level in order to control the particles flow.

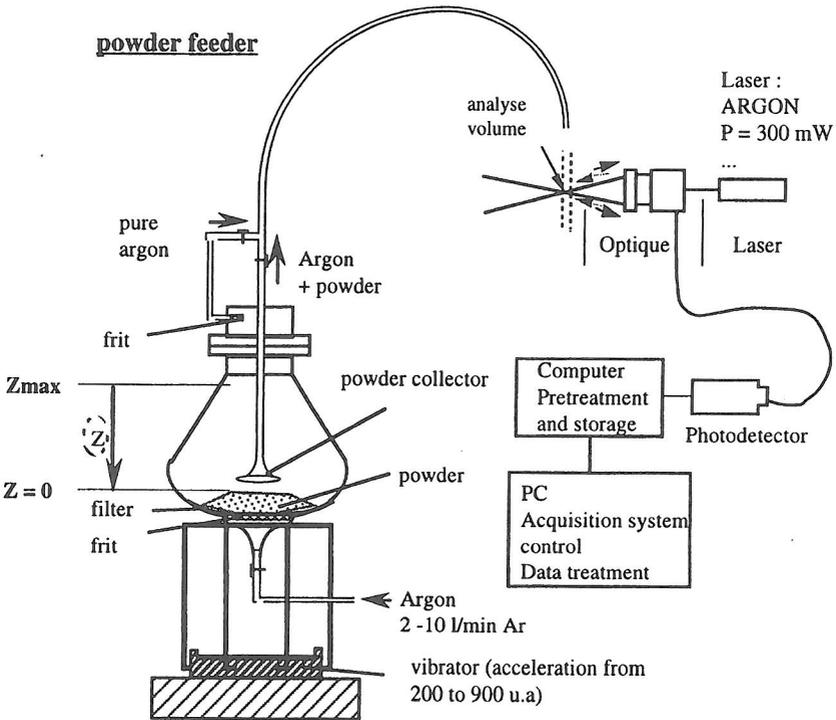


Fig.2 Scheme of the powder feeder and back scatter signal detection

### 2 The Laser Doppler Anemometer

The measurement are obtained by means of LDA system with a 300 mW argon-laser source (focal length 54 mm) across an ellipsoid analyse volume ( $1 \text{ mm}^3$ ) of interference fringes ( $3,23 \text{ }\mu\text{m}$ ). The back scatter signal is picked up with a photo multiplier and treated with a dynamic analyser 58N10 (fig.2).

The results are graphically expressed by means of the velocity distribution and the time series signal. The first one gives the quantity of particles as a function of the velocity. In the second one, the particle signal is pecked-up as a function of the time (fig.3).

Regularity of particles feeding is calculated as the quotient of the average time needed for a particle to pass through the ellipsoid and the mean difference with this time. The powder flow rate is calculated as the quotient of the quantities of particles and the experience time.

### 3 Results and discussion

Different experimental parameters have been studied by means of LDA: the particle size (30-50  $\mu\text{m}$ , 50-80  $\mu\text{m}$  and 80-125  $\mu\text{m}$ ); the fluidized bed gas flow (4 and 8  $\text{l}\cdot\text{mn}^{-1}$ ); the vibrating frequency (300-350 and 650-800 u.a); the height of the collector above the powder level (0-5 cm).

The results show a linear relationship between the particle velocity and their granulometry for an size higher than 40  $\mu\text{m}$ . They also show that the particle velocity is equal to the gas one for a diameter smaller than 15  $\mu\text{m}$  (fig.4). Furthermore, the powder flow values increase when the gas flow increases and when the collector reference position decreases. The regularity of the injection increases with a rising granulometry and a rising collector position. It also increases when the gas flow values and the vibration frequency decreases. It corresponds to a compromise between the regularity and the powder flow.

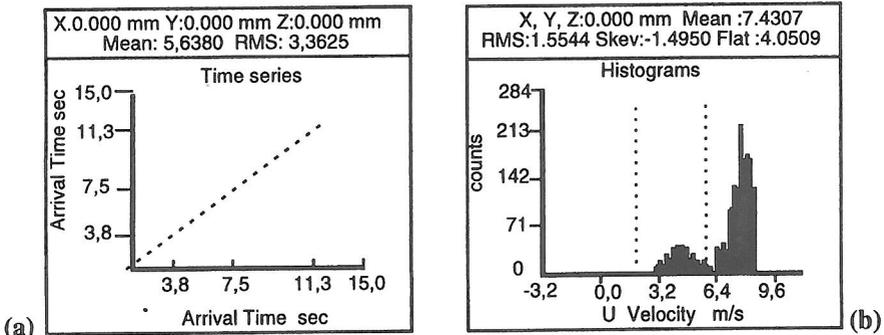


Fig.3 Measurement at the exit of the injector: regularity of the injection (a); histograms of the velocity distribution (b).

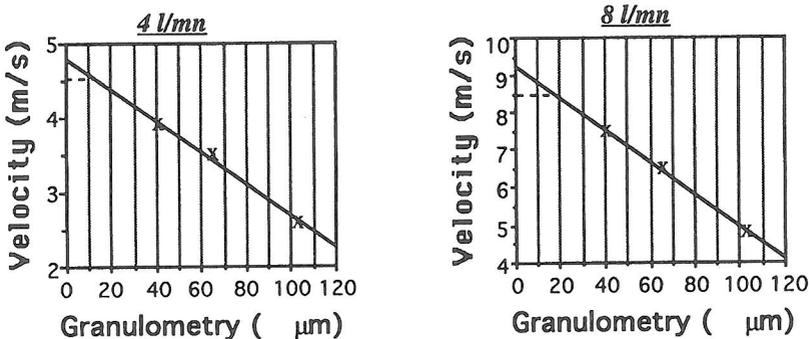


Fig.4 Particle velocity at the exit of the injector expressed as a function of their granulometry and the fluidized bed gas flow (4 l/mn and 8 l/mn).

Finally the best experimental parameters are selected as shown in fig.5.

Granulometry ( $\mu\text{m}$ )	30-50				50-80				80-125	
	Ar powder ( $\text{l.mn}^{-1}$ )	4		8		4		8		8
z (cm)	0		1		0		1/2		0	
Acceleration (u.a.)	300	650	300	650	320	720	320	720	350	800
$V_{\text{granu}}$ (m/s)	3,9		7,5		3,5		6,5		4,8	
Mass (g)	$7,81 \cdot 10^{-8}$		$7,81 \cdot 10^{-8}$		$2,98 \cdot 10^{-7}$		$2,98 \cdot 10^{-7}$		$1,31 \cdot 10^{-6}$	
$E_c = 1/2 \cdot m \cdot v^2$ (MeV)	4000		14000		12000		40000		95000	
$E_c$ [ $\text{J} \cdot 10^{11}$ ]	59		439		365		1259		3018	

Fig. 5 Parameters of the powder distribution chosen for the injection in the plasma.. Determination of the particles kinetic energies.

### III PARTICLE SPEED MEASUREMENTS INTO A PLASMA JET BY LDA

#### 1 Measurements into the plasma jet

The main difficulty for LDA measurements into a plasma jet is due to the plasma electromagnetic radiation (4 MHz) which disturbs the back scatter signal. To avoid this problem, it is necessary to choose an other optical frequency. Furthermore the particles flow by means of a cold fluidized gas, so it's difficult to inject them in the viscous plasma flow. In order to avoid this problem it's necessary to place the powder injector exit close to the first spire coil (fig.7).

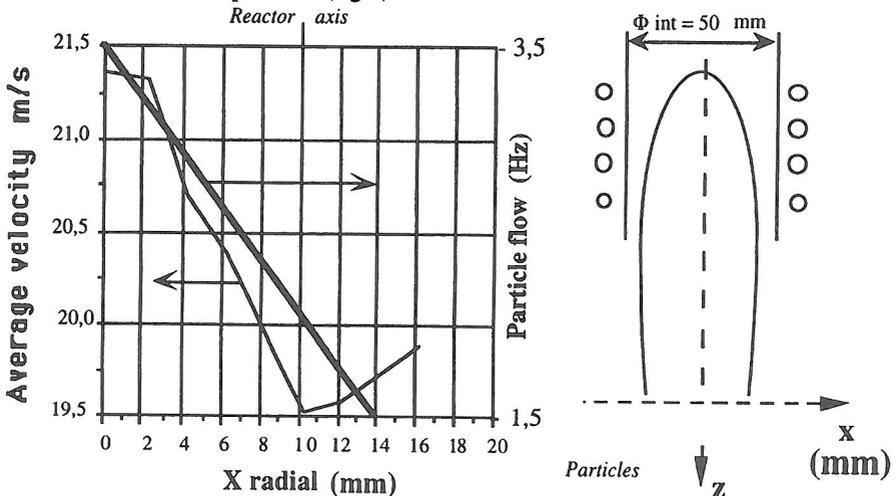


Fig.6 Radial profile of the particle velocities in the plasma:  $v = f(x)$  (measurement conditions: argon plasma,  $30 \text{ l.mn}^{-1}$  axial,  $40 \text{ l.mn}^{-1}$  annular, 20 kW).

The first results (fig.6) show that the particles go out the injector at 5 m/s and are accelerated up downstream the induction coil region and reach the maximum velocity in the axial flow (21 m/s)(fig.6). The plasma jet length is 30 cm and the mean residence time of the particle into is 15 ms.

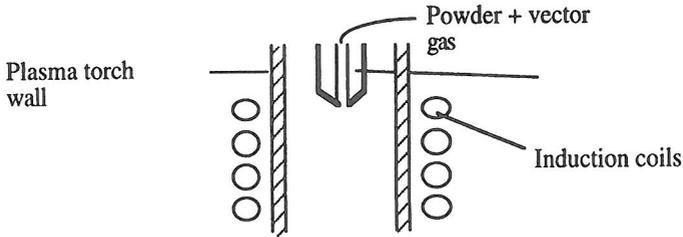


Fig.7 Location of the powder injector up the induction coils.

### 2 Modelling correlation

The residence time is estimated by a model developed in the laboratory [5] (fig.8) and the microscopic analysis of the deposition (fig.9 a et b). The model shows that a particle of silicon having 100 μm immersed in an argon-plasma with a mean temperature of 5000 K, needs a resident time of 5 ms for a total fusion.

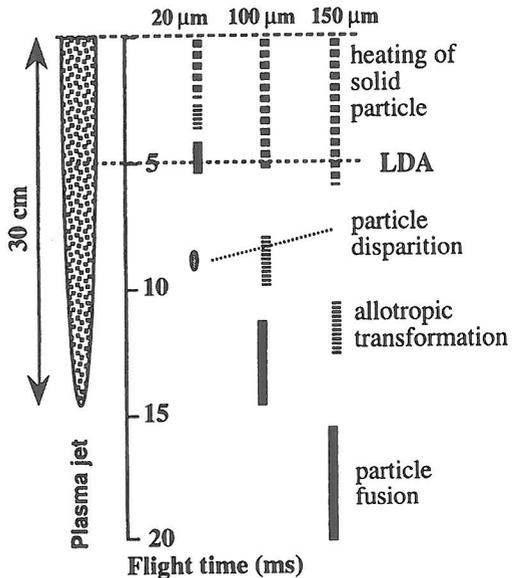


Fig.8 Modelling of the thermal evolution (melting and evaporation) of the injected particles in the plasma.

### 3 Deposition analyses

The particles collected in the lower part of the reactor after the plasma treatment have an average diameter lower than the starting one. For this reason we can conclude that their is a strong evaporation phenomena in agreement with the conclusions of the model (fig. 9). However, some of them are not evaporated but, these sprayed particles on the target, point out that those which have a size close to 100 μm are fully melted.

scale : 100  $\mu\text{m}$

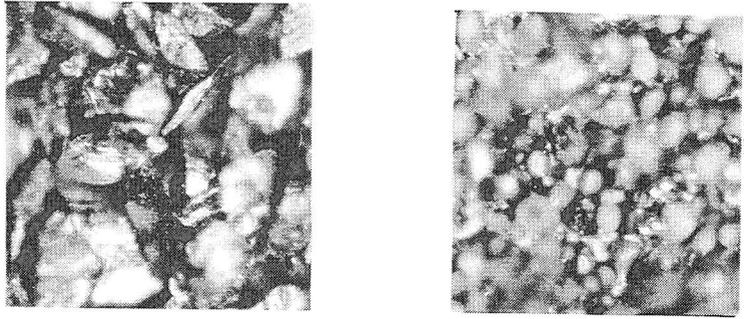


Fig.9 a and b

Silicon particles before (a) and after (b) plasma treatment.

#### IV CONCLUSION

LDA measurements show a good correlation between modelling and experimental results. The melting kinetic will be improve with few percentages of hydrogen in argon flow and the partial evaporation of particle in the plasma will be followed by atomic emission spectroscopy. This online spectroscopic diagnostic will be combined with a new reactor. Together with LDA, its will allow us to measure the influence of the plasma chemical composition on the mechanism of purification of the particle by a selective evaporation of the impurities during their flight.

#### REFERENCES

- [1] M.Vardelle, A.Vardelle, P.Fauchais. "The effect of air entrainment on the behaviour of powders in plasma spraying: comparative study under ambient atmosphere and inert environment." 8th I.S.P.C. Tokyo, Japan, 1987
- [2] M.Vardelle, A.Vardelle, P.Fauchais. "Powder vaporisation under plasma conditions experimental investigation of the vapour cloud surrounding a single particle." 8th I.S.P.C. Tokyo, Japan, 1987
- [3] D.Morvan, R.Combes, J.Erin, I.Cazard-Juvernat, J.Amouroux. "Diagnostic of the Chemical Phenomena near a material melted by a thermal Plasma. Correlation between experimental measurements by AES, a theoretical model and the final chemical measurements of the material." Proceeding of Seminar on Heat and Mass Transfer under Plasma Conditions to be Held, 4-8 July 1994 Izmir Turkey [To be published]
- [4] D.Morvan, J.Erin, I.Cazard-Juvernat, J.Amouroux. "Effects of hydrogen on heat transfer from RF Argon plasma to silicon and purification process. Characterisation of the material." Proceeding of 3rd European Congress on Thermal Plasma Processes, 19-21 Septembre 1994, Aachen Germany
- [5] P.Humbert, D.Morvan, J.Amouroux. "Modelisation of heat and mass transfer between a particle and a thermal plasma. Journal of high Temperature Chemical Processes 1 (1992) pp 57-76
- [6] I.Cazard-Juvernat, O.Bartagnon, B.Pajot, D.Morvan, J.Amouroux. "Oxygen elimination and new electronic properties of silicon due to a treatment by thermal plasma (Ar-H<sub>2</sub> or Ar-He). [Paper presented is this meeting]

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