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# Plasma spraying and extractive metallurgy: comparisons between mathematical modelling and measurements and between application and development

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Abstract - After a brief description of the industrial developments of thermal plasmas in the fields of thermal spraying and extractive metallurgy, the state of the art of our knowledge in the following subjects is reviewed:

- modelling of the plasmas, plasma transport properties (LTE or two temperatures conditions) and cold gas mixing
- modelling of the plasma-particle momentum and heat transfer
- measurements of the plasma jet temperatures and velocity distributions
- measurements of the particles in flight in the plasma jet : velocity, surface temperature, trajectory, size evolution...
- correlations between measurements and calculations

## INTRODUCTION

The formation of a protective coating by plasma spraying a stream of molten metal or ceramic particles was developed during the sixties. The major advantage of plasma compared to flame is the higher particle velocity obtainable (up to 500 m/s) and the high temperatures achieved (more than 10 000 K) making possible to melt the most refractory materials. Rapid solidification by plasma deposition (cooling rates up to $10^7$ K/s) combines melting, quenching and consolidation in one single operation and such cooling rates result in grain sizes of 0.25 to 0.5 $\mu$m for metals and alloys {1 to 7}. However the quality of the coatings obtained depends strongly on one hand on the heat and momentum transfer between particles and plasma (the particles must be melted upon impact) controlling also the chemical reactions of the particles with their environment during their flight as well as their decomposition and on the other hand of the heat transfer control to the substrate and deposit while spraying (grain sizes, cracks to relax the stresses, metastable phases, contacts between the lamellas...).

For a long time, industrial development of plasma sprayed coatings has been mostly empirical, the physical and chemical understanding of the phenomena lagging far behind. However with the important development of plasma sprayed deposits in aeronautics, mechanics, nuclear engineering, electrical engineering etc. {3 to 7} a better understanding of the phenomena involved is needed to improve the quality of the coatings as well as the spraying yields; the properties of such desposits required by industry being more and more sophisticated.

This improved knowledge of the phenomena is also needed with the industrial development of plasma jets or of transferred arcs for :

- melting and purification {8 to 10} by transferred arcs ($100 \leqslant P \leqslant 1\ 000$ kW) struck in a controlled atmosphere chamber between a water cooled crucible and a cathode; the material to be treated being introduced as particles, pellets, rods, hollow pieces etc.
- heating : steel billets {11}, ladle {11,12}, blast air injected at the tuyeres of blast furnaces {13,14} ($0.5 \lesssim P \lesssim 3$ MW/torch).
- scrap melting with power levels reaching now 7.5 MW/torch {8, 9, 11, 15 to 21}
- extractive metallurgy, still at its very beginning, using mostly transferred arcs {22 to 25} but with high power pilot plants for example in South Africa (Middelburg 10.8 MW, Mintek 3.5 MW, Semancor 8.5 MW)
- plasma reformer for direct reduction, mainly developed by SKF {8, 9, 26} either for direct reduced iron production (Plasmared at Hofors, 70000 T/year) or for smelt reduction (Plasmazinc at Landskrona, 70000 T/year),

The aim of this paper is to make a brief review of the state of the art in the fields of modelling and measurement of plasma jets, seeded or not, with solid particles and to try to underline where the lacks are and what is still needed.

## 2-PLASMA PROCESSES MODELLING

### 2-1-Plasma flows

In spite of the intensive research effort that has been devoted over the last thirty years to the study of electric arcs, the mathematical modelling has developed slowly due to the difficulties encountered in the analytical description of the electrode regions {27}. These regions are characterized by their small dimensions (of the order of $10^{-2}$ mm), very steep temperature gradients and important non equilibrium effects {27 to 30}. It seems that the arc is capable of producing a vast variety of different phenomena induced by minor changes of the mechanical properties of the electrodes, of their geometry, of small impurities at their surface. If at the moment the cathode phenomena are still far to be well understood, specially for the emission phenomena of cold cathodes {31, 32} (cylindrical copper electrodes widely used now in arc gas heaters {11, 13, 14}), some phenomena have been emphasized for cold anodes with argon as plasma gas {29} such as negative anode drop, instead of the positive one generally assumed, and the strong non equilibrium effects, phenomena allowing to understand better the heat transfer. However, what happens when the anode material evaporates (of primary importance for melting, smelting, extractive metallurgy, welding with transferred arcs) has to be studied.

The experiments of Tsantrizoo et Gauvin {33} with a transferred argon arc on a molten copper anode have shown an important evolution of the voltage as soon as the anode evaporates. Very recent spectroscopic measurements performed in Limoges {34} with a TIG striked onto an iron plate show that two plasma regions can be distinguished : a quasi "pure argon" plasma region in L.T.E. and a "metallic vapor plasma region" near the anodic molten bath. In this latter region, a strong disequilibrium between the "argon temperature" values and the neutral iron excitation temperature values is observed. The results suggest that the amplitude of the thermal transfer to the workpiece depends largely on the efficiency of the elementary processes that govern the energy exchanges between the "argon" plasma and the metallic "vapor" plasma within the arc plasma column.

The electrode phenomena must take into account the elementary processes (collisions and non equilibrium effects) as well as the flow problems (how to model the balance between the drag force due to the cold gas flow near the walls and the electromagnetic force due to the bending of the plasma column when the arc strikes at the nozzle anode of a plasma torch ) and it is at the moment far too complex to be included in the flow models. The flow models {35} are developed either for the arc column {36 to 41} or for plasma jets exiting the nozzle {42, 43}. The main assumptions of such models are that the plasma is in L.T.E., the jet is steady and posesses cylindrical symmetry, radiation transfer is negligible as well as compressibility effects (except for supersonic plasma flows as those used for spraying under reduced pressure {44}). Two types of approaches are then possible :

- the plasma jet is supposed to be laminar and the flow is described by the Navier-Stokes equations, continuity and conservation of energy (see for example {28, 39})
- for high Reynolds numbers, obtained with rather low temperature plasmas ($T < 6000$ K), the dependent variables are decomposed into mean and fluctuating parts and the resulting equations are then time averaged to produce the equations for the evolution of the mean quantities. Usually {42} density weighted averaging is used and the mean flow equations are closed by assuming gradient diffusion for the turbulent correlations and using $K-\varepsilon$ turbulence model for the Reynolds stresses (see for example the resulting equations in {36 or 43}).

The governing equations are then put in a finite difference form (typically using a 15 x 15 or 15 x 20 grid {36}) and solved numerically using iterative procedures due to the coupled nature of the equations {45}. These numerical solutions are classified in parabolic and elliptic. The parabolic and much simple case corresponds to a one-way behavior (the flow is called boundary-layer-type) i.e with no influence of the downstream on the upstream because axial convection dominates axial diffusion. The forward marching solutions algorithms such as those of GEMMIX program {45} extended to plasmas by numerous authors, greatly reduces computational time. Most of the plasma jets or transferred arcs are relevant of this type of parabolic solutions. However when a plasma jet exiting in a pipe is considered, for example to heat the blast air in the tuyeres of a blast furnace, recirculation problems, participating to the heating of the whole gas, have to be considered and then elliptic solutions must be used for example with the SIMPLE program {46}. In this case downstream boundary conditions are needed. Typically these are taken zero axial gradients but if the boundary is not far enough downstream, the jet decay rate will be affected and the computational domain should therefore, be increased, until the results are unaffected, but the grid must remain fine enough to resolve steep gradients {42}. In such heating flows with elliptic models the problem of heat transfer to the surrounding walls is also of primary importance. For the conductive-convective fluxes, the choice of the proper grid and of the velocity distributions in the viscous area near the wall, in the transition region and the fitting with their fully turbulent area far from the wall is complex {47} and the radiative fluxes cannot be neglected {48}.

Other problems arise from the boundary conditions and for example up to now the surrounding gas pumped by the fast plasma flow has always been supposed to be of the same nature as the plasma gas, thus avoiding the calculation of complex transport properties available only for gases such as Ar, He, $N_2$, $H_2$, $O_2$ and for some mixtures $N_2$-$O_2$, $N_2$-$H_2$, Ar-$H_2$ {49}. These transport properties are very sensitive to the choice of the interaction potentials {50}. The anode evaporation also requires the knowledge of transport properties plasma gas-metallic vapor and up to now results are only available for Ar-Cu {51} and Ar-Fe {52}, the unknown interaction potentials being assumed to be hard core ones. Chen and his co-workers {53} have also demonstrated that the symmetrical injection of a cold gas into the plasma jet modifies strongly the equilibrium, the temperature discrepancy between electrons $T_e$ and heavy particles $T_h$ being most severe at the location of cold flow injection (cold argon in an argon plasma). This effect is introduced in the governing equations by splitting the energy equation in two: one for the electrons and one for the heavy particles with a closure equation relating $T_e$ to $T_h$ via elastic collisions. The corresponding thermodynamic properties are calculated using the Modified Saha Equations (MSE) method introducing the ratio $\Theta = T_e/T_h$ and the transport properties by extending the work of Devoto developed at equilibrium. However recently Bonnefoi {54} has proposed a new definition of the diffusion forces in a two temperatures (2 T) model and a different approach of the reaction term {55}. It is worth noting that the formulas developed for the transport properties in a 2 T model make use of the collision integrals calculated at equilibrium {54}. Aubreton {56} has demonstrated that this approach is valid for $\Theta \leqslant 3$ (typical ratio for atmospheric thermal plasmas where a cold gas is injected or for reduced pressure plasmas down to about 40 Torr). For $\Theta > 3$ it is necessary to use a kinetic approach for which the nature of the gas (monoatomic or diatomic) has a great importance {56}. However for heat transfer between a plasma and particles the integrated values of the thermal conductivity are not really affected by the differences between the two methods. Up to now these 2 T calculations have been developed for Ar, $H_2$, $O_2$ and Ar-$H_2$, Ar-$O_2$ mixtures {57, 58}.

Moreover the injection of a cold gas chemically different from the plasma gas, can induce important chemical reactions such as those obtained when injecting cold oxygen into a nitrogen plasma {59}. With the high temperature gradients encountered in thermal plasmas (heating rates up to $10^9$ K/s, cooling rates up to $10^8$ K/s due for example to the fast expansion of the jet) the kinetic calculations should be included in the flow models thus making them very complex with the stiffness of the solutions of the kinetic equations {59, 60}. That is why, up to now, none of these calculations have been developed in the general case ; the first results obtained being limited to flows with uniform radial velocity and temperature distributions {59, 60}.

At last it should be underlined that all the developments of the flow models are 2D but that most practical problems are 3 D (particle injection for spraying, plasma torch blowing in the tuyere of a blast furnace etc.) and up to now only big companies such as Westinghouse in U.S.A., E.D.F. in France {61} have developed 3 D models for gas heating, but simplified models where the plasma properties are kept constant to reduce the computing time.

### 2-2-Plasma particle momentum and heat transfer

In view of the paper of Pfender {62} in this issue, we will just give here a few indications about the problems involved. Due to the importance of the thermal treatment of powders in plasma torches and furnaces a considerable attention has been given to the plasma particle momentum and heat transfer (see for example the references given in {35, 63 to 66}). These works underline the necessity to take into account corrections terms or integrated thermal properties for the steep gradients in the boundary layer round the particles {62, 63}, non continuous effects for particles smaller than about 10 m at atmospheric pressure {67, 68}, turbulent dispersion {62}, charging effect {62}, evaporation effect {64}. For given temperatures and velocity distributions of the plasma jet, the trajectory and temperature history of individual particles, assumed to be spherical, are calculated. However in practice what is needed is the statistical behavior of the injected particles which have size and velocity distributions. For a given injector diameter and a given carrier gas flow rate the particles will have (due to their size distribution) different injection velocities and even with the proper injection velocity, the particles passing near the injector wall will have their velocity tending to zero. That is why {69} the trajectories and temperature histories of the particles must be calculated for different sizes and velocities and the results averaged according to the starting distributions and particles flow rates. Moreover, when the particle mass flow rate increases too much the particles start to cool down the plasma jet as well as when small particles evaporate consuming a large amount of energy {70} and this has to be included in the calculation program rending it very heavy. At last one has to underline that all these calculations neglect the cold gas injection that would requires a 3 D calculation to take into account its effect on the plasma flow.

## 3-MEASUREMENTS AND COMPARISON WITH MODELLING

### 3-1-What is needed

Quite a lot of data are needed :
- temperature and velocity distributions of the plasma flow as well as non-equilibrium effects
- concentration of the different species excited or not
- trajectory, velocity, surface temperature of the particles

to compare the calculated distributions with the measured ones, to have reliable models and to obtain relevant data either for the particles (drag and Nusselt coefficients accounting for the various phenomena envolved) or for the plasma gas itself (diffusion coefficients, chemical rates etc.).

### 3-2-Plasma jet measurements

#### 3-2-1-Temperature distributions

What can be reached easily {71, 72} with emission spectroscopy, is the excitation temperature (through atomic spectra), the electron density (through line profiles of Stark enlarged lines), the rotational and vibrational temperatures (through rotational spectra), but the electron temperature has to be calculated with the help of the preceeding data {73}. Of course such measurements give averaged values (for times of a few tens ms) masking the fluctuations of the arc. However, due to the steep radial gradients, Abel's inversion has to be performed. That is why almost all the measurements have been performed for axially symmetric jets and a big effort has been made to automatize these measurements, by moving the plasma {74}, using rotating mirrors {75}, displacing rapidly a metal strip into the jet {76}, devices allowing rather fast measurements for atomic line intensities, by using 2D optical multichannel analysers (OMA) {77} allowing fast measurements of rotational spectras (up to 40 lines) or of line profiles. It is worth to notice that rotational spectra will give temperatures in the range 3500 – 9000 K about and atomic lines in the range 8000 K – 13000 K, while ionic lines are between 15000 and 21000 K and the precision is about 10 %. The temperature ranges encountered in the various plasmas are the following : for heating 3000 – 9000 K about, for spraying 3000 – 12000 K, for transferred arcs 7000 – 18000 K and one has to remind that, according to the fast variation of the volumic emission coefficients with temperature, a range of three decades about is accessible for a given set of the measurement device corresponding to $\Delta T$ = 4000 K at the maximum.

In a transferred argon arc at atmospheric pressure, where the electron density is rather high ($n_e \sim 10^{22}$ $e/m^3$), the measured excitation temperatures {78} are in reasonable agreement (within 15 %) with the calculated distributions.

However for a nitrogen d.c. plasma jet where cold nitrogen is introduced symmetrically, the measurements have shown {72, 79} that :
- when increasing the cold gas flow rate, the temperature isocontours (measured from rotational spectra corresponding, due to the fast relaxation translation-rotation, to the heavy particles temperature) are pinched and lengthened
- the cooling of the fringes result in a fast diffusion of the electrons from the plasma center to the periphery of the jet
- the population of the levels close to ionization limit is thus no more in thermal equilibrium with the one of the levels close to the resonant one
- the diffusion phenomena, very important in this case, have to be included in the models where they have been neglected up to now

Of course these first results obtained for the cold gas injection emphasize the non-equilibrium effects already taken into account in the models {53} but where diffusion effects have to be introduced. It would be also necessary to develop the measurements in non-symmetrical plasmas where these effects are probably enhanced. Non-symmetrical Abel's inversion are now possible with the use of computers on line to account for the quantity of data to be treated. They have already been developed in a simple case for thermal plasma jets {80}, the use of OMA being very promising for such measurements.

If the problems of investment costs are not considered, CARS technique {81} could probably be used in thermal plasmas to measure the heavy species temperature, the extension of the temperature range (developed for combustion up to 3000 K), being quite possible (up to 7000 K). The advantage of CARS over emission spectroscopy is the possibility to obtain a very good spatial resolution (avoiding the problems of Abel's inversion), an important signal (even in the plume of the jet) and probably to measure the temperatures in plasmas seeded with particles (in combustion flame the temperature seems to be almost insensitive to the presence of soot particles).

Laser induced fluorescence (LIF) gives signals proportional to the number density difference $\Delta N_u$ of the upper level due to laser pumping and the problem is to relate $\Delta N_u$ either to the temperature or to the density of the lower level (often a fundamental level or a metastable one). Such relationships in the general case are obtained through the matrix density {82} and it is only in particular conditions, generally not fullfilled in thermal plasmas, that the approximation of the rate equations can be used {83}. That is why in thermal plasmas such measurements can give only relative values {84}, however very instructive, showing for example that NO in its fundamental state is produced mainly in the periphery of a nitrogen d.c. plasma jet where cold oxygen is injected symetrically. However LIF is the only mean to obtain informations about reaction or quenching routes via experiments performed at reduced pressure (0.1 to 10 Torr ) in a flowing afterglow where the different species about to react are excited through collision transfers with various metastable atoms {85}.

### 3-2-2-Velocity distributions

The methods using the Doppler shift of the lines are limited to supersonic jets at low pressure (below 50 Torr) {72} and up to now only the seeding of the jet with small particles, which velocity is measured by LDA has been used in thermal plasmas {74}. However the precision of the measurements is questionable due to measurement difficulties with small particles and to the problems of momentum transfer between plasma and small particles (Knudsen effect among others underlined by the results presented at Montreal {86} for 40 $\mu$m particles in plasmas at 50 Torr).

Such uncertainities in the measurements may partly explain the discrepancies between measured and calculated temperature and velocity distributions in thermal spraying jet {42, 43}. It is necessary to emphasize the importance of having a reliable velocity distribution at the nozzle exit to obtain correct results with the models developed for spraying plasma jets where particle heating occurs in a few tens of millimeters. That is why a recent method of resonant Doppler velocimetry with alkali atoms seeded in flames {87} might be very interesting if it is possible to extend it to thermal plasmas.

Of course the problem is different when one wants to heat a cold gas injected round the plasma jet, the influence of initial velocity and temperature distributions of the plasma being almost negligible for the distributions obtained at distances comprised between 6 and 10 times the pipe diameter.

### 3-3-Particle measurements
### 3-3-1-Velocity

LDV is the main technique used {71, 72, 88} ; it allows high spatial resolution (less than 1 $mm^3$) and high temporal resolution (down to 5 ns). Among the different detection devices, only counters and frequency trackers are able to associate a velocity with a single given particle. To perform the measurements in the plasma core itself requires on one hand the use of monochromators with bandpass round 1 Å in order to eliminate, as much as possible, the light emitted by the plasma and on the other hand either to increase the power level of the laser or to increase the dimensions of the measurement volume by performing it within an angle close to the laser beam direction to obtain the maximum emission of the scattered light.

### 3-3-2-Surface temperature

The up-to-date technique is that of discrete in flight color pyrometry, first developed with absolute flux measurements {71, 74} which precision was poor (the result depends on the emission coefficient of the particle and on its diameter which varies as soon as evaporation starts). Recently {72, 89, 90} this technique has been developed by measuring the ratio of fluxes emitted at two wavelengths (two color pyrometry) thus eliminating the problem of the diameter and reducing the one of unknown emission coefficients (assumption of grey body). Actually such measurements, performed in volumes of Ø = 160 $\mu$m, l = 15 000 $\mu$m, are statistical measurements and give in fact surface temperature distributions. However it is important to underline that, whatever will be the future technique, it will never be possible to perform the measurements in the core of the plasma jets, heating zone of the particles and where their flux cannot overcome the plasma flux as long as their temperature is not high enough (more than 2 200 K for 20 $\mu$m particles about).

### 3-3-3-Particle trajectories and concentrations

The number of particles travelling at different locations in the plasma jet may be measured by counting, for a given time, the pulses resulting from the light scattered by the particles passing through a focussed laser beam. A measurement volume of less than $10^{-3}$ $mm^3$ is achievable {91, 72}. The particle mean trajectory is determined from the position of maximum concentration of the particles. It is worth to notice that, even with very narrow size distributions ($Al_2O_3$ particles 18 ± 3 $\mu$m) injected with the optimum velocity, the trajectory distribution is large : 20 mm dowstream the injector, it covers about 1/3 of the surface of a plasma jet "slice" {72}.

### 3-3-4-Particle sizes

Combined measurements of velocity and size of particles are achieved by extended laser doppler anemometers : the amplitude and modulation depth of LDA signals depend on the size of the scattering particles, the optical properties of the particle material, the wavelengths of the employed laser radiation, the angle between the two incident beams and the size and location of the receiving aperture {92}. These sizing methods are essentially {88, 93} the visibility and pedestal calibrations methods where size and velocity measurements are achieved on the same signals and the power calibrations methods for which the optical probes for velocimetry and sizing are different.

## 3-4-How these measurements are correlated to calculation

First one has to remark that the measurements on the particles and on the plasma flow are performed separately and second that, to get reliable informations, the measurements on the particles (v, $T_S$, flux) should be performed at same location and time. If previous comparisons between the calculated and measured velocities gave a reasonable agreement {94, 95} (within 15 %) for measurements along the axis, the discrepancies between measured and calculated temperatures have been reduced with the two color pyrometry {89}. However the last measurements {69} underline the importance of various effects : heat propagation for ceramics, evaporation, Knudsen etc. but also the necessity to make statistical calculations of the trajectories and thus velocities, surface temperatures and diameters to compare measured distributions with calculated ones.

## CONCLUSION

Plasma spraying has now a wide industrial development and users require more sophisticated properties of the deposits. If plasma remelting, purification and extractive metallurgy are still in their infancy, the first results obtained are promissing and raise a great interest for industry. That is why a better knowledge of the phenomena involved is needed specially for modelling various plasma devices configurations taking into account mixing, chemical reactions, non-equilibrium effects if possible using 3D configurations. However due to the complexity of the models and to the various assumptions the results are meaningless if they are not compared with measurements and a great effort has to be done to computerize all the devices already available to start a systematic study of the mixing of a cold gas with a plasma, of the reduced pressure spraying devices, of the particles injection and behaviour, of the heat transfer to the walls or electrodes, of the chemical kinetic.

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