

## Shielded open air plasma spraying of reactive materials

***Citation for published version (APA):***

Houben, J. M., & Zaat, J. H. (1976). Shielded open air plasma spraying of reactive materials. In *ITSC : 8th international thermal spraying conference : proceedings, Miami Beach, Florida, September 28 - October 1, 1976* (Vol. 1, pp. 78-85). American Welding Institute.

***Document status and date:***

Published: 01/01/1976

***Document Version:***

Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

***Please check the document version of this publication:***

- A submitted manuscript is the version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
- The final author version and the galley proof are versions of the publication after peer review.
- The final published version features the final layout of the paper including the volume, issue and page numbers.

[Link to publication](#)

***General rights***

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal.

If the publication is distributed under the terms of Article 25fa of the Dutch Copyright Act, indicated by the "Taverne" license above, please follow below link for the End User Agreement:

[www.tue.nl/taverne](http://www.tue.nl/taverne)

***Take down policy***

If you believe that this document breaches copyright please contact us at:

[openaccess@tue.nl](mailto:openaccess@tue.nl)

providing details and we will investigate your claim.

# SHIELDED OPEN AIR PLASMA SPRAYING OF REACTIVE MATERIALS

By J. M. Houben and J. H. Zaat  
Eindhoven University of Technology, The Netherlands.

## SUMMARY

A shielding device for open air plasma spraying has been developed, tested and put to practice. The device mainly consists of a diverging tube in which two fluid streams are established: the plasma jet and a coaxially flowing stream of primary shielding gas, thus producing a coherent gas coverage of the transport trajectories and the substrate spray area. Test results show that the oxygen content of the spray gases within the gas-covered area of the substrate can easily be reduced to 0,2 percent. Spray deposits of Mo do not contain more oxygen than the spray powder. Sprayed deposits on the basis of Sn-Sb show good performance as bearing material; the oxygen content of these deposits vary from 0,018-0,04 weightpercent.

## 1 INTRODUCTION

Since a free plasma jet for spraying purposes has a predominantly turbulent character, such a jet will always involve the problem of mixing with the ambient air. Oxygen will penetrate the plasma gases as reported by Hasui<sup>1</sup> e.a. The spray particles will absorb the oxygen and will be oxidized during their flight from torch to substrate. This oxygen contamination is one of the main problems related to conventional plasma spraying of reactive materials such as molybdenum.

To avoid the contamination, adequate inert gas protection must be given to the particles and the deposit during the spray operation. Up to the present time, the most reliable method of producing near by oxygen free spray deposits of reactive materials such as molybdenum, makes use of a special chamber into which the workpiece to be treated is placed<sup>2,3,4</sup>. The chamber may be evacuated and operated under vacuum or may be filled with inert gas to give the required high degree of protection. Equipment of this type however is expensive and limits the size of the part to be treated. The work partly reported here deals with the development of "shielded open air" technique for which there is a need. As far as the authors are familiar to this object, two other investigators<sup>5,6</sup> have recently published about this technique. Both authors make use of a

coaxially jet shielding tube. From their reports it is obvious that their methods have been successfully put to practice. Although the shielding device to be discussed in this paper is related to those which are mentioned above it differs essentially from these methods. Jackson's device attains the shielding effect by blowing a considerable amount of shielding gas around the jet, which is not the case in this one and Okada et al. use an extra, what they call "shield cover" with a diameter of 200 mm, which also is absent in the following construction.

## 2. DIFFUSER TYPE SHIELDING TUBE<sup>+</sup>

A typical representation of such a device is outlined in fig. 1. The ambient air is not permitted to penetrate the plasma stream by enclosing the jet and the transport way of the spray particles within a diffuser. Inside this diffuser two coaxially flowing fluid streams are established: the plasma jet and a primary shielding gas stream surrounding this plasma jet. The shielding diffuser is essentially a diverging tube whose purposes are to increase the pressure of the fluid streams by reducing the flow velocity and to stabilize a coherent stream of gas coaxial with the plasma jet. This gas stream has to cover a certain workpiece area with a shielding atmosphere from which the air is excluded. A diffuser essentially converts the kinetic energy of the streaming gases into potential energy, i.e. pressure. There are two main problems involved when applying a diffuser as a plasma spraying shielding device.

- a. The spray particles must leave the torch coaxially with the plasma. An excentric spray jet will cause a deposit of spray material inside the shielding tube, blocking the proper operation of the device. Besides the divergence angle of the spray jet, if coaxially with the plasma, must be restricted. These problems have been solved first<sup>7</sup>.
- b. The pressure recovery in a diffuser will not quite reach a hundred percent. There is an irreversible pressure drop, compared to the theoretical ideal recovery according to Bernoulli's law, because the inlet area of

<sup>+</sup> Patent pending.

the diffuser, does not correspond to the exit bore of the anode. Besides there are losses due to wall friction causing another pressure drop compared to the ideal case of non viscous flow.

If the flow inside the diffuser were uniform over the cross section, then these pressure drops would not be of great importance for the diffuser's performance. However the friction losses have considerable effect especially on the boundary layers, the more so, because a diffuser for spraying purposes has got a short length and a relatively large divergence angle. Flow separation can occur and a flow pattern as sketched in fig. 1, curve a must be considered as being usual for such a short diffuser if not sufficient primary shielding gas is supplied.

So to avoid the penetration of air into the spray gases along path "a", additional measures must possibly be taken.

The unfavourable effects due to fluid deceleration along the diffuser wall previous to flow separation, can be overcome basically in two ways:

- a. By removing the decelerated fluid elements along the wall before flow separation and subsequently, penetration of air occurs. To realise this, suction slots can be installed as shown in fig. 2.
- b. By acceleration of the slow fluid particles. This can be done effectively by blowing in secondary shielding gas along slots in the diffuser wall, also show in fig. 2. Details of the action of suction and blowing on the boundary layer profile are given in fig. 3.<sup>8</sup>

### 3. TEST MEASUREMENTS

Shielding tubes provided with these design features have been put to practice.

At this moment experimental data based on a tube working with primary shielding gas but not yet fitted with suction and secondary injection slots, are available.

A schematic of the experimental set up for testing is presented in fig. 4.

The total pressure distribution over the centerline of a cross section was obtained for varying primary shielding gasflow rates by using a water cooled Pitot-tube, connected to a manometer, basically existing of a tilted glass tube filled with alcohol.

The velocity profile can be deduced from the total pressure and additional static pressure and temperature measurements. Typical results are shown in fig. 5 and 6.

From fig. 5 it appears that in absence of a substrate in front of the tube and without supplying of shielding gas in the tube a reverse flow of air along the wall inside the tube will occur.

The plasma jet works like an injection pump and no shielding effect is achieved.

From fig. 6 it appears that a positive total pressure profile at the exit plane of the tube can be obtained by placing the tube in spray position in front of the substrate at a distance of 5 mm and additional supplying of 50 lmin<sup>-1</sup> N<sub>2</sub> as primary shielding gas. Without or with to small an amount of shielding gas, the flow can not be kept attached to the tube wall and flow reversal and subsequential air penetration occurs.

Oxygen measurements in the spray gases show the effect of the shielding tube performance if the proper quantity of primary suppletion gas is supplied, see fig. 7. The oxygen concentration in vol.% is measured over a centerline in the gascovered area of the substrate at a 5 mm stand off distance between tube and substrate. Curve a and b in the presence of the substrate, curve c in absence of the substrate. It is clear from this figure that both the nearness of the substrate and the suppletion gas flow rate are of crucible importance for the performance of the device.

### 4 SPRAY RESULTS.

The diffuser type shielding tube has been put to practice. Molybdenum and, among others, a Babbitt bearing alloy called V 738 were sprayed. Table 1 summarizes some spray results for Mo. The emphasis was placed on the reduction of the oxygen content in the sprayed deposit. As appears from table 1 the oxygen content of the deposit did not surpass the original content of the spray powder. The microhardness figures of the deposit show approximately 12% increase. The deposit efficiency is 10% less than is the case when spraying without shielding tube. Probably this phenomenon is due to the fact that no oxidation occurs, so the extra heat generation from oxidation is absent as may be concluded from the heat content figures. Converting the lowest figure 920 kJ/kg into the temperature of the Mo particles, it turns out that they are just about melting, whilst the heat content of 1000 kJ/kg means that approximately 37% of all particles are completely molten. Good adhesion with high deposit efficiencies can be expected when all the particles are completely molten as is the case when the heat content surpasses the level of 1190 kJ/kg. So, the conclusion can be drawn, that the power level for a successful application of this shielding device must be increased compared to the non-shielded way of spraying. The structure is nevertheless of a non permeable type. It shows epitaxy at the grain boundaries, see fig. 8a and 8b.

Table 1: Spray results for Mo

Column I : Using centerline injection  
System I : According to reference 7

Column II: Using the same centerline injection system combined with shielding tube and a shielding gas consumption of  $50 \text{ lmin}^{-1} \text{ N}_2$

Torch-exit to substrate distance mm	I	II	Spray-material Mo
$O_2$ % (weight) of deposit	1,61	0,155	0,149
	1,59	0,098	0,142
	1,59	0,135	0,147
		0,094	
		0,11	
		0,11	
Vickers Hardness $H_{V25}$ kgf mm <sup>-2</sup> at distance x from substrate			
x $\mu\text{m}$			
20	488	237	
50	677	306	
80	673	289	
110	657	370	
140	627	342	
170	503	306	
200	525	285	
230	548	351	
260	572	240	
290	627	333	
320	599	317	
350	588	336	
Average	588	309	275
Deposit efficiency average %	83	73	
Heat content <sup>a</sup> of spray particles kJ/kg	1110-1260	920-1000	

a. Power levels from 9-17 kW over the electrodes.

Quite another and rather unusual type of plasma spray material which has been sprayed using the shielding tube is the V738 material. It is a typical soft bearing material and it was of interest how the application of a shielding device would work out on the structure and mechanical properties of sprayed deposits with respect to their application as a bearing surface.

For reasons of good heat conduction, the bearing material should have only a slight degree of porosity, if any. A good adhesion to the substrate may be considered as a basis for good overall performance of the bearing. It has been tested by Chalmer's method. Also of great importance is the macrohardness with

respect to the maximum load that can be applied. Another aspect is the dynamical behaviour of the bearing. So, sprayed bearings were tested on a M.A.N.-test device which consists basically of a rotating shaft surrounded by loaded half shells of the bearing to be tested. The inner diameter of the bearing is 50 mm. The load and the number of revolutions of the shaft can be varied. The test run is carried out in the following way. In the beginning a small load of  $2 \text{ Nmm}^{-2}$  is applied. Then a stepwise increase of shaft's rotation speed takes place until a radial velocity of  $20 \text{ ms}^{-1}$  is attained. Then the load is stepwise increased until break down of the bearing occurs. Table 2 summarizes some test results of shielded plasma sprayed bearings. These results can be compared with figures for cast bearings of the same material. The chemical composition is given in table 3.

Table 2. Test results of shielded plasma sprayed and cast V738 bearings.

Test	Cast	Sprayed
Maximum dynamical load acc. to M.A.N. test $\text{Nmm}^{-2}$	18,60	19,60
Adhesion strength $\text{Nmm}^{-2}$	90	92 91
Brinell Hardness 2,5/31,25/180 $\text{Kgf.mm}^{-2}$	25,9 25,5 25,5 24,8 25,0	27,3 27,9 27,8 24,6 28,4

The deposits were sprayed onto tinned steel substrates. Some main spray parameters are summarized in table 4.

From table 2 it is obvious that the sprayed bearings behave mechanically very well. As to the chemical composition (table 3) it is clear that most elements are "burned out" somewhat during the spray operation.

Although can be expected that this loss of elements has got an adverse effect on the strength of the deposits, it appears that the strength is not affected really. Probably the very fine structure as shown in fig. 8c and 8d compensates for possible reduction of strength based on chemical composition. The structure shows fine irregular shaped crystals consisting of a solid solution of Cu-Sn and Ni-Sn, fine cubic shaped crystals of Sn-Sb solid solution and a matrix with high Sn content.

Table 3 . Chemical composition of spray powder V738 and shielded plasma sprayed deposit.

Element	V738	Weight percent		
		size range $\mu\text{m}$		
		53-75	75-105	105-150
Cd	1,2	0,13	0,12	0,19
Cu	5,5	5,24	4,97	4,93
Sb	12	10,5	11,3	11,8
Ni	0,3	0,3	0,28	0,28
As	0,5	0,35	0,35	0,35
O <sub>2</sub>	0,03	0,04	0,032	0,018
Sn	Balance			

Table 4. Main spray parameters.

size range $\mu\text{m}$	53-75	75-105	105-150
Deposit efficiency %	59	75	75
Heat content of spray particles $\text{kJkg}^{-1}$	460	465	410
Current	300-330 Amp		
Voltage	23- 24 volt		
Arc gas	30 $\text{lmin}^{-1}$ , Argon		
Shielding gas	50 $\text{lmin}^{-1}$ , N <sub>2</sub>		
Spray distance	130 mm		
Powder flow rate	40 $\text{gmin}^{-1}$		
Substrate temp.	110-150 and 150-200 <sup>o</sup> C		
Deposit thickness	~ 6 mm.		

Porosity has been extremely reduced.

Further investigations will be directed on the following items:

- reduction of primary shielding gas consumption
- Application of suction and secondary injection slots in order to force the flow kept attached to the wall
- combination of cooling and shielding gas supply

- increasing of the stand off distance between shielding tube and workpiece.
- avoiding any deposition of rebounded spray particles or metal and metal oxide vapor on the inner tube wall.

#### 5. CONCLUSION

A plasma spraying shielding device for open air technique has been developed and successfully put to practice. Using this device in combination with centerline injection, deposits of reactive materials which are ductile and poor in oxygen content and porosity can be produced at powerlevels of approximately 8-17 kW over the electrodes.

#### 6. ACKNOWLEDGEMENT

The authors are grateful to mr. G.G. van Liempd for his assistance in the experimental work.

#### 7. REFERENCES

1. Hasui.A, Kitahara.S, Fukushima.T. Some properties of plasma jet for spraying. Trans. of Nat. Res. Inst. for Metals vol.7, no. 5 (1965) . pp 186-194.
2. Glätzle . R., Benesovsky. F., Das plasma-spritzen von Wolfram. Metall Vol.24. Aug. 1970. H. 8, pp 823-829.
3. Steffens. H.D. Production and properties of homogeneous deposits of special materials with high oxygen affinity. Proc. 8th Internat. Metal Spraying Conference, 1970, Paris.
4. Muehlberger. E. A high energy plasma coating process. Proc. 7th Internat. Metal Spraying Conference, 1973, London.
5. Okada. M., Maruo. H. New Plasma spraying and its application. British Welding Journal Aug. 1968. pp 371-386.
6. Jackson.E. Method for shielding a gas effluent. U.S. patent 3 470 347. Sept. 1969.
7. Houben.J.M. Some remarks on plasma spraying powder injection techniques. Paper to be presented at the 8th Int. Thermal Spraying Conf. 1976. Miami.
8. Käppeli. E. Strömungslehre I-Blaue TR-Reihe Heft 113, Hallwag Verlag. Bern und Stuttgart.
9. Hilgers.W. Brochure Goldschmidt Informiert 2/70 nr. 11 pp 2-24.

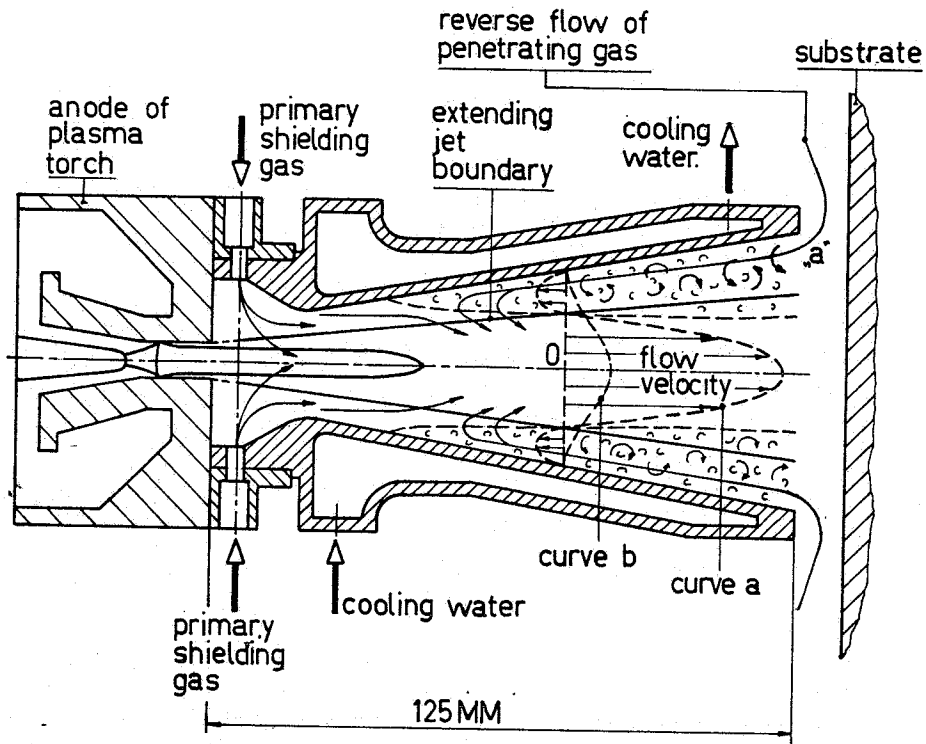


FIG.1

Schematic of diffuser type shielding tube with velocity profiles.  
Curve a : velocity profile if shielding gas is insufficiently supplied.  
Curve b : overall positive velocity profile if shielding gas is sufficiently supplied.

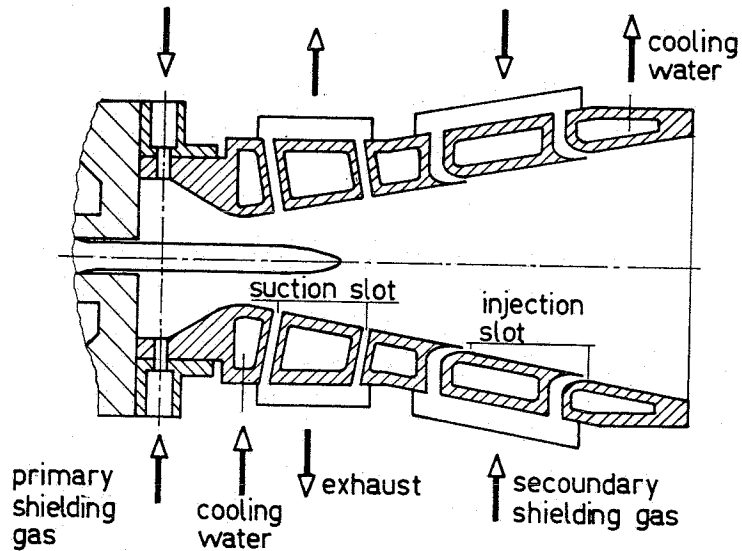
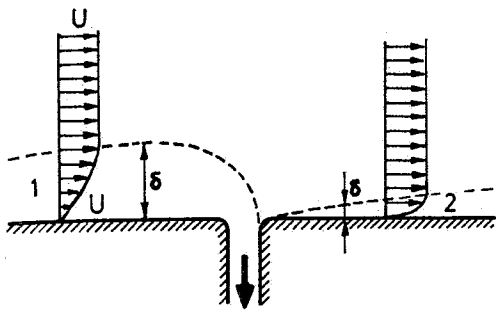
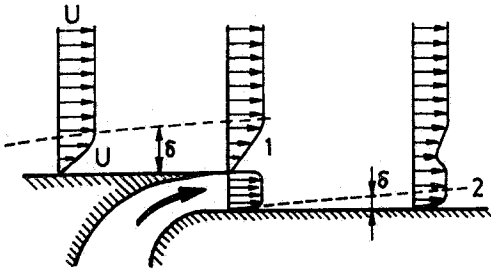


FIG. 2

Diffuser type shielding tube provided with suction and secondary gas injection slots.



a



b

FIG. 3

- a. Influence of suction on velocity profile of the boundary layer.
- b. Influence of secondary gas injection on velocity profile of the boundary layer.

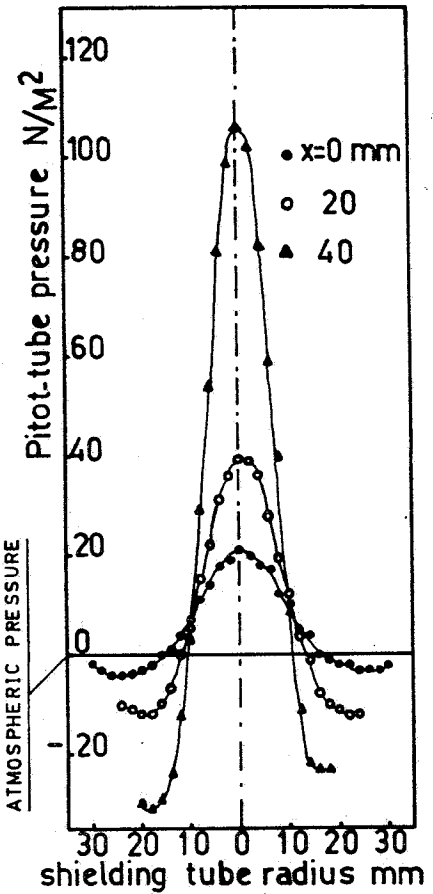


FIG. 5

Total pressure profiles over cross-section of plasma jet enclosed by diverging shielding tube without supply of primary shielding gas and in absence of a substrate in front of the exit plane.

X = Internal axial location starting from tube's exit plane.

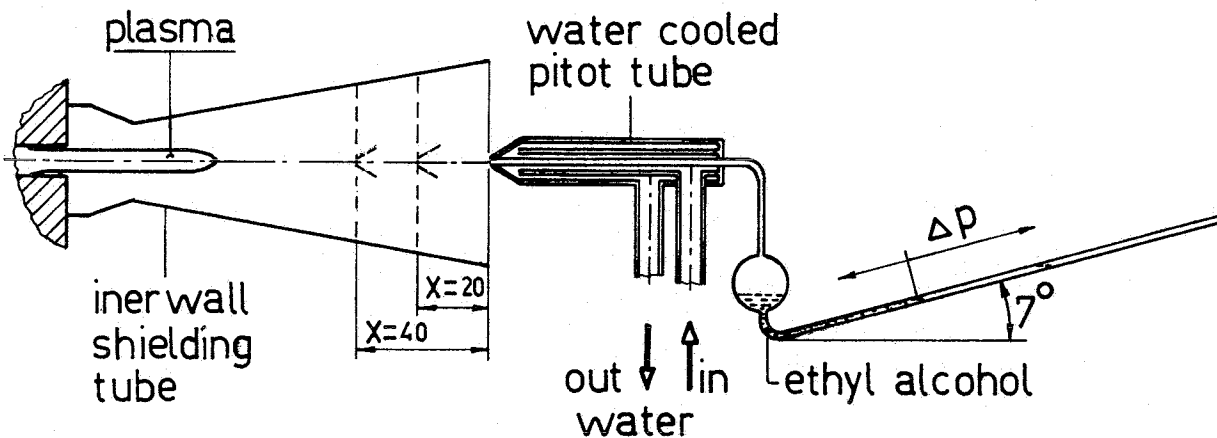


FIG. 4 Schematic of the experimental set up for tube test.

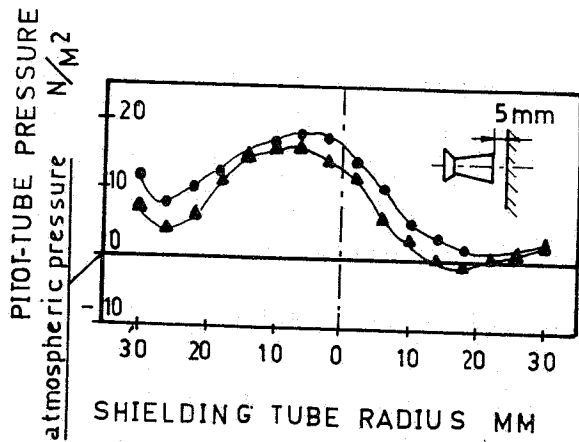


FIG. 6

Pressure profiles at exit plane of shielding tube in front of a flat substrate when primary shielding gas is supplied:

- 50  $\text{lmin}^{-1}$   $\text{N}_2$
- 25  $\text{lmin}^{-1}$   $\text{N}_2$

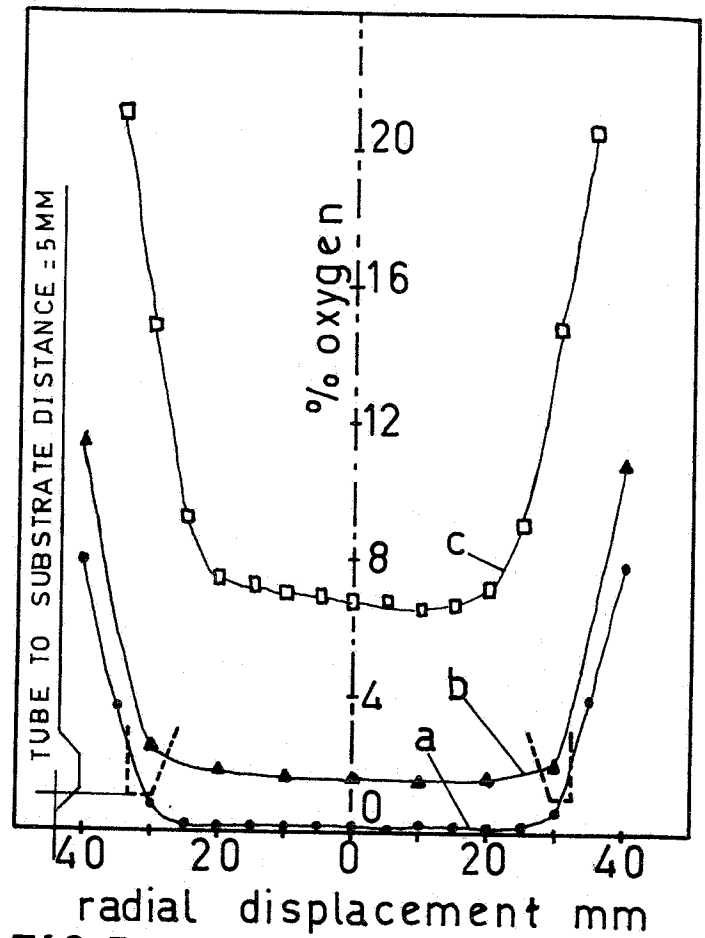
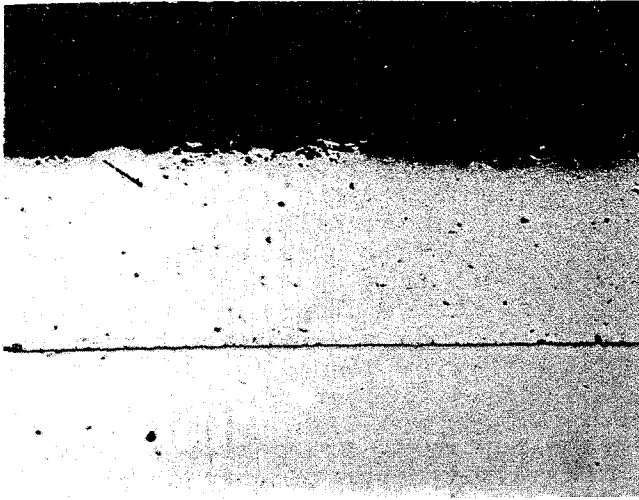


FIG. 7

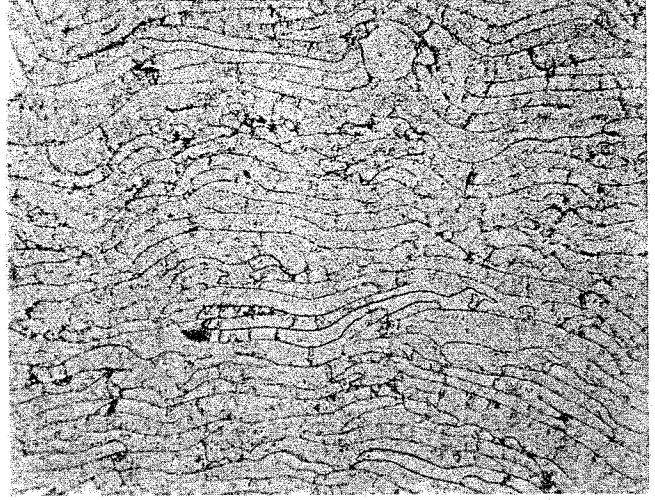
Oxygen content of the spray gases in the covered area of the substrate.

- Curve a: -distance tube to substrate 5 mm  
-primary suppletion gas rate  $50 \text{ lmin}^{-1} \text{N}_2$
- b: -distance tube to substrate 5 mm  
-without suppletion gas
- c: -primary suppletion gas rate  $50 \text{ lmin}^{-1} \text{N}_2$   
-without substrate.

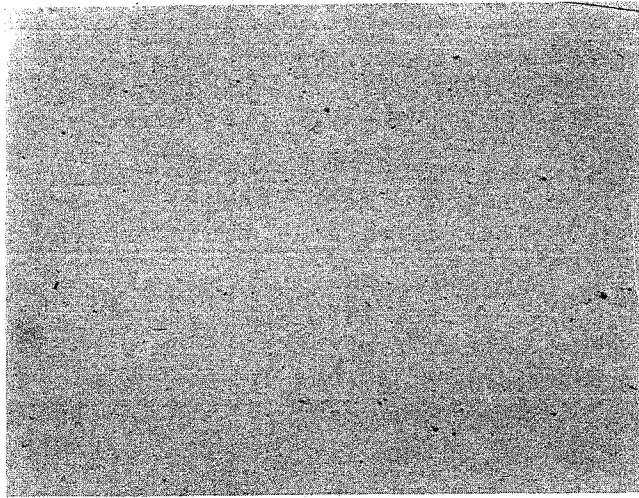




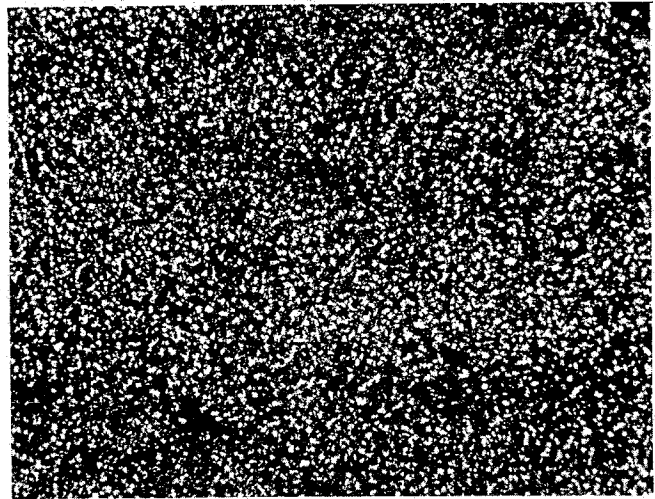
a — 156  $\mu$ m



b — 22  $\mu$ m



c — 166  $\mu$ m



d — 22  $\mu$ m

**FIG. 8** Plasma sprayed deposits using diffuser type shielding tube.  
a. Molybdenum  
b. Detail of fig. a  
c. V738-Babbit alloy  
d. Detail of fig. c.