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ABSTRACT

TiB_2 cermets can be obtained from titanium bearing alloys and boron via SHS processes. The synthesis of TiB_2-Fe materials produced by the reaction of titanium-iron alloys with boron was particularly studied.

TiB_2-Fe parts containing a fine microstructure were fully densified by hot isostatic pressing previously synthesized powders. Their mechanical properties and wear resistance were determined.

Thick TiB_2-Fe coatings were also obtained by the plasma spray synthesis (PSS) process, i.e. by plasma spraying micropellets comprising the reagents. The microstructure and wear resistance of these coatings are also presented.

1. INTRODUCTION

Exothermic reactions can be used as a means for the synthesis of ceramic or metallo-ceramic materials. The term "self propagating high temperature synthesis" is usually referred to a process involving the simultaneous synthesis and shaping of materials. This process is claimed to reap advantage from the heat generated during the reaction.

In this work, TiB_2-Fe cermets were synthesized through the exothermic reaction of ferrotitanium with boron. Parts were produced by hot isostatic pressing powder coming from the reaction products. Coatings were also obtained by the so-called Plasma Spray Synthesis (PSS) process consisting in synthesizing and depositing these TiB_2-Fe cermets in a one step.

This work aimed at characterizing the mechanical properties and abrasion resistance of TiB_2-Fe parts and coatings which have been done through SHS.

2. SYNTHESIS OF TiB_2-Fe

2.1 Starting Materials

TiB_2-Fe materials were synthesized through the reaction of loose ferrotitanium and amorphous boron powders. Two commercial ferrotitanium powders were used in this work as starting materials (Table I). The ferrotitanium denoted "A" is particularly impure. It contains manganese and possesses a very high oxygen content. The ferrotitanium denoted "B" is purer than "A" and its oxygen content is only 0.06 wt%. However, its titanium content is lower than "A". The contituents identified by X-Ray diffraction analysis in these ferrotitanium powders are given in Table II. Large amounts of FeTi and Tiβ were found in both ferrotitanium powders. A complex iron-titanium oxide (Fe_xTi_yO) is also present in large amount only in ferrotitanium "A". Figure 1 depicts the particle size distribution of the ferrotitanium and boron powders.

2.2 Reaction

The ferrotitanium powders were mixed with amorphous boron powders in stoechiometric proportions according to the following reaction:

$$FeTi + Ti + 4B \rightarrow 2TiB_2 + Fe \qquad (1)$$

Figure 2 shows the thermograms recorded during the reaction of ferrotitanium "A" and ferrotitanium "B" with boron respectively. In both cases, the initiation of the reaction starts at about 700°C.

2.3 Reaction Products

X-Ray diffraction analysis of reaction products showed that large amounts of TiB_2 and Fe were synthesized. Low intensity peaks corresponding to Fe_2B were also observed (Fig. 3). The oxygen content in reaction products was measured (Table III). It appears that the high oxygen content in reaction products could be related to oxygen present in the starting ferrotitanium powders and it can be stated that the synthesis of TiB_2-Fe does not result in purer products. As pure are the reagents as pure would be the reaction products.

3. CERMET PARTS MADE BY HOT ISOSTATIC PRESSING

TiB_2-Fe powders were first synthesized by igniting a mixture of loose ferrotitanium and boron powders in a furnace at 1100°C under argon atmosphere. The resulting friable reaction product was ground to - 200 Mesh. Rectangular green shapes were obtained by cold pressing the powder. These green compacts were encapsulated in low carbon steel capsules and then hot isostatically pressed.

The effect of HIPing temperature on the density of parts is shown in Figure 4. It can be seen that nearly full dense specimen can be produced at a temperature of 1300°C. The typical microstructure of HIPed parts made with ferrotitanium "A" and "B" are shown on Figure 5 respectively.

The mechanical properties of these parts have been evaluated. Figure 6 shows the influence of the total fractional porosity (P) of TiB_2-Fe cermets on their flexural strength (σ) can be described by the two following expressions:

$$\sigma = 708 \text{ exp. } (-8.44P) \text{ (for ferrotitanium A)}$$

$$\sigma = 1100 \text{ exp. } (-12P) \text{ (for ferrotitanium B)}$$

where σ is expressed in MPa and P by a dimensionless number. From the relations (2) and (3), one can predict that fully dense parts synthesized from ferrotitanium A and B should possess flexural strengths of 708 MPa and 1100 MPa respectively. The higher flexural strength of cermets synthesized from ferrotitanium B is related to their lower TiB_2 volume content than that made from ferrotitanium "A". The purity of starting reagents particularly oxygen content could be also responsible for this difference in flexural strength.

Abrasive wear measurements were carried out on HIPed TiB_2-Fe cermets in accordance with the ASTM G-65 method. The data from Table IV point out that these cermets possess a significant abrasion resistance as compared to cemented carbide.

4. PLASMA SPRAYED-SYNTHESIZED COATINGS

TiB_2-Fe coatings can be directly obtained via the Plasma Spray Synthesis (PSS) process. In this process schematically illustrated in Figure 7, micropellets comprising the reagents (ferrotitanium and boron) are first introduced into the flame. They are heated up until the reaction took place. Finally, the reaction products containing TiB_2 and iron are propelled onto a substrate to build up the desired coating.

For this purpose, the micropellets (agglomerates) were prepared by agglomerating with the suitable agglomeration technique ferrotitanium and boron powders. Other elements such as nickel could be added at this step to increase the performance of the resultant coatings. Figure 8 shows the appearance of these micropellets before spraying. Conventional plasma spraying equipment was used to process the agglomerates.

Figure 9 shows the typical microstructure of a SHS TiB_2-Fe coating. By selecting the appropriate spraying parameters, the PSS process may lead to dense coatings with Vickers microhardness values higher than 1550 Kgf mm^{-2} (100g load). It could be pointed out that the microstructure of these coatings is considerably finer than those of parts produced by HIPing (Figs. 6 and 7).

As dense parts, the abrasive wear resistance of these coatings was measured in accordance with the ASTM G-65 method. As shown in Table V, the performance of TiB_2-Fe coatings approach that of the expensive high energy WC-Co coatings. Furthermore, by comparing Table IV and V, it could be pointed out that the abrasion resistance of coatings is as good as that of HIPed dense parts. The refinement in microstructure following high solidification rates and the volatilization of low temperature oxides during spraying could explain this better performance.

5. CONCLUSION

Cermet powders, parts and coatings produced by SHS reactions are not only interesting because they can be of lower cost. Processing materials via SHS reactions may be considered as a powerful means to produce materials with unique microstructures. HIPed TiB_2-Fe cermets, exhibited mechanical properties and abrasion resistance far beyond those obtained previously with the same materials. Thick composite coatings synthesized by this convenient Plasma Spray Synthesis (PSS) process are, of course, more interesting than fragile thin films.

TABLE I

CHEMICAL ANALYSIS OF FERROTITANIUM AND BORON

MATERIAL	ELEMENT (wt%)									
	Ti	C	Si	Al	Mn	Cr	B	Mg	O_2	Fe
Ferrotitanium A	73.69	0.28	0.23	0.94	6.64	–	–	–	2.74	Bal.
Ferrotitanium B	62.9	0.09	–	0.96	0.86	0.64	–	–	0.06	Bal.
Boron	–	0.03	–	–	1.92	–	94–96	1 max.	1.38	–

TABLE II

CONSTITUENTS PRESENT IN FERROTITANIUM POWDERS

	MAJOR	MINOR
Ferrotitanium A	FeTi, Ti_β, Fe_xTi_yO	
Ferrotitanium B	FeTi, Ti_β	Fe_xTi_yO

TABLE III

OXYGEN CONTENT IN REACTION PRODUCTS *

STARTING MATERIALS	O_2 (wt%)
Ferrotitanium A + Boron	1.62
Ferrotitanium B + Boron	0.78

* Synthesized by heating at 1100°C in argon.

TABLE IV

ABRASION RESISTANCE OF HIPed TiB_2-Fe CERMETS

MATERIAL	VOLUME LOSS (mm^3)	ABRASION RESISTANCE FACTOR (1000/Volume loss)
SAE 1020	294	3.4
TiB^2-Fe*	14.8	67.6
WC-6.8 wt% Co	4.2	238

* Synthesized with ferrotitanium A HIPped:
1200 s at 1250°C and 180 MPa.

TABLE V

ABRASION RESISTANCE OF SHS TiB_2-Fe COATINGS

COATING	VOLUME LOSS (mm^3)	ABRASION RESISTANCE FACTOR (1000/Volume loss)
TiB^2-Fe* (fine agglomerates)	18.9	52.9
TiB2-Fe* (coarse agglomerates)	15.8	63.3
WC-11 wt% Co (Mach II parameters)	13	76.9

* Ferrotitanium B was used.

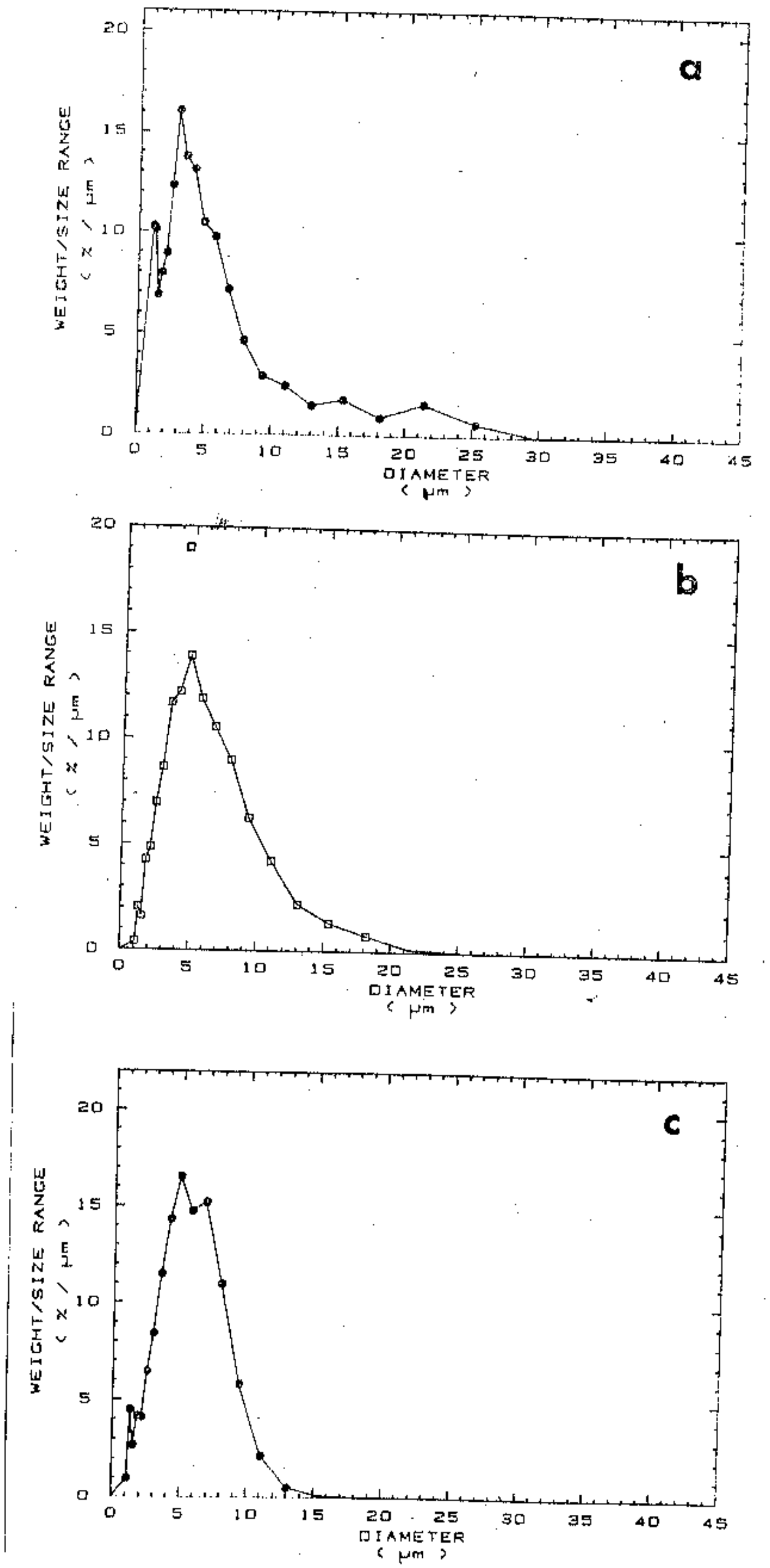
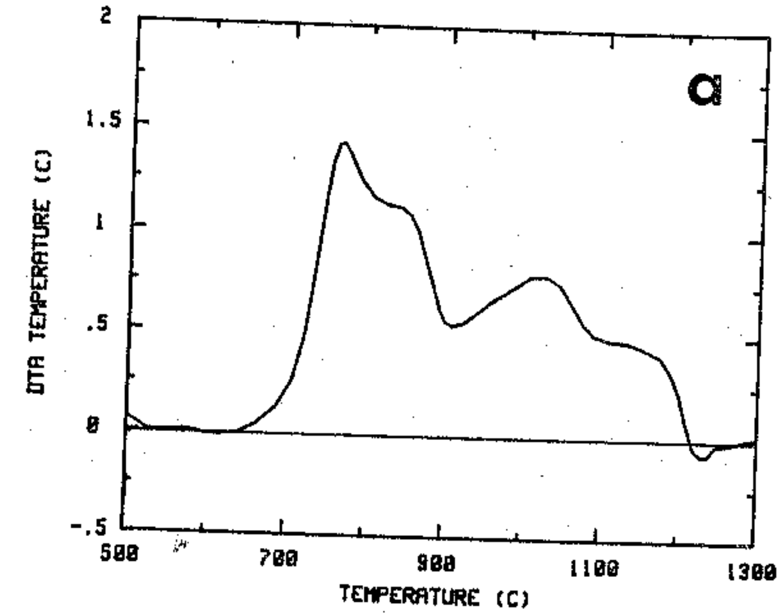


Fig. 1 - Particle size distribution of:
(a) Ferrotitanium A; (b) Ferrotitanium B; (c) Boron.



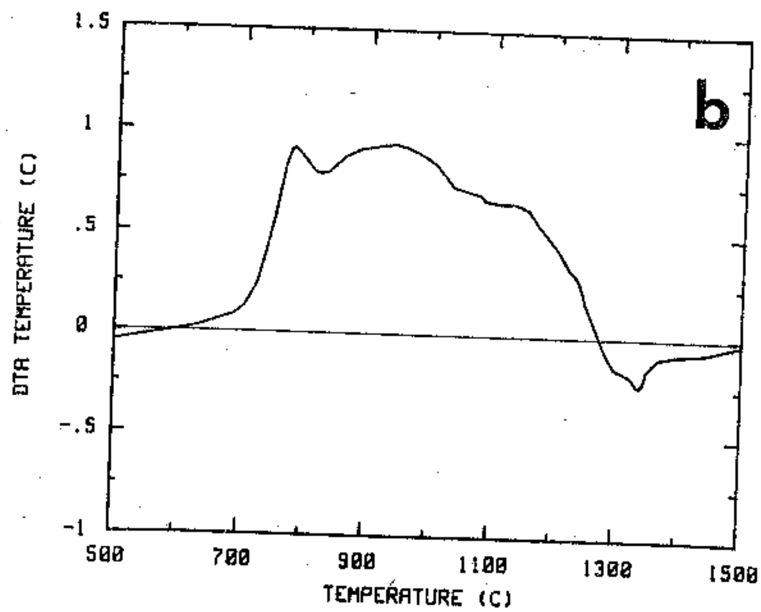


Fig. 2 - Differential thermal analysis of:

(a) Reaction of ferrotitanium A with boron;
(b) Reaction of ferrotitanium B and boron.

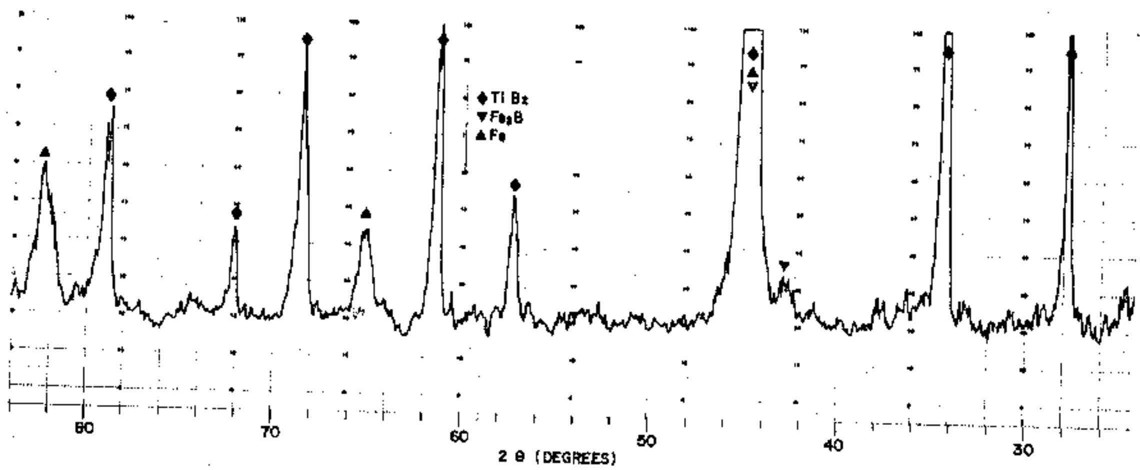


Fig. 3 - X-Ray diffraction pattern of reaction products (from ferrotitanium B and boron).

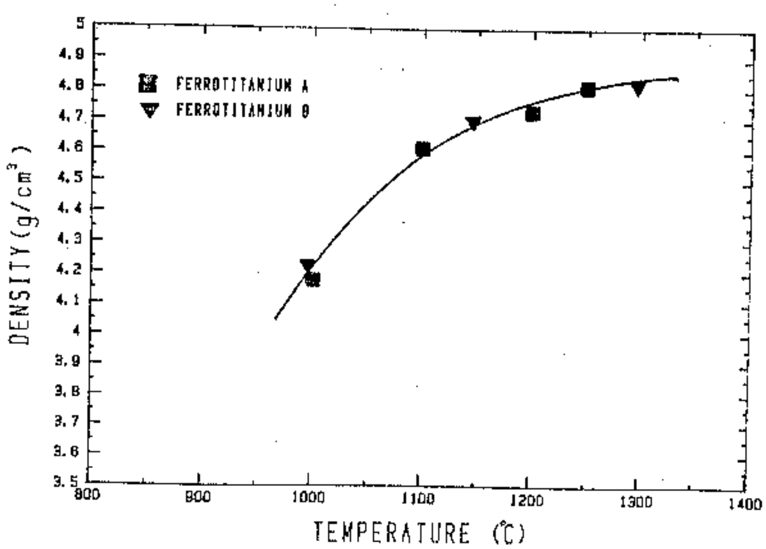


Fig. 4 - Effect of HIPing temperature on density of parts.

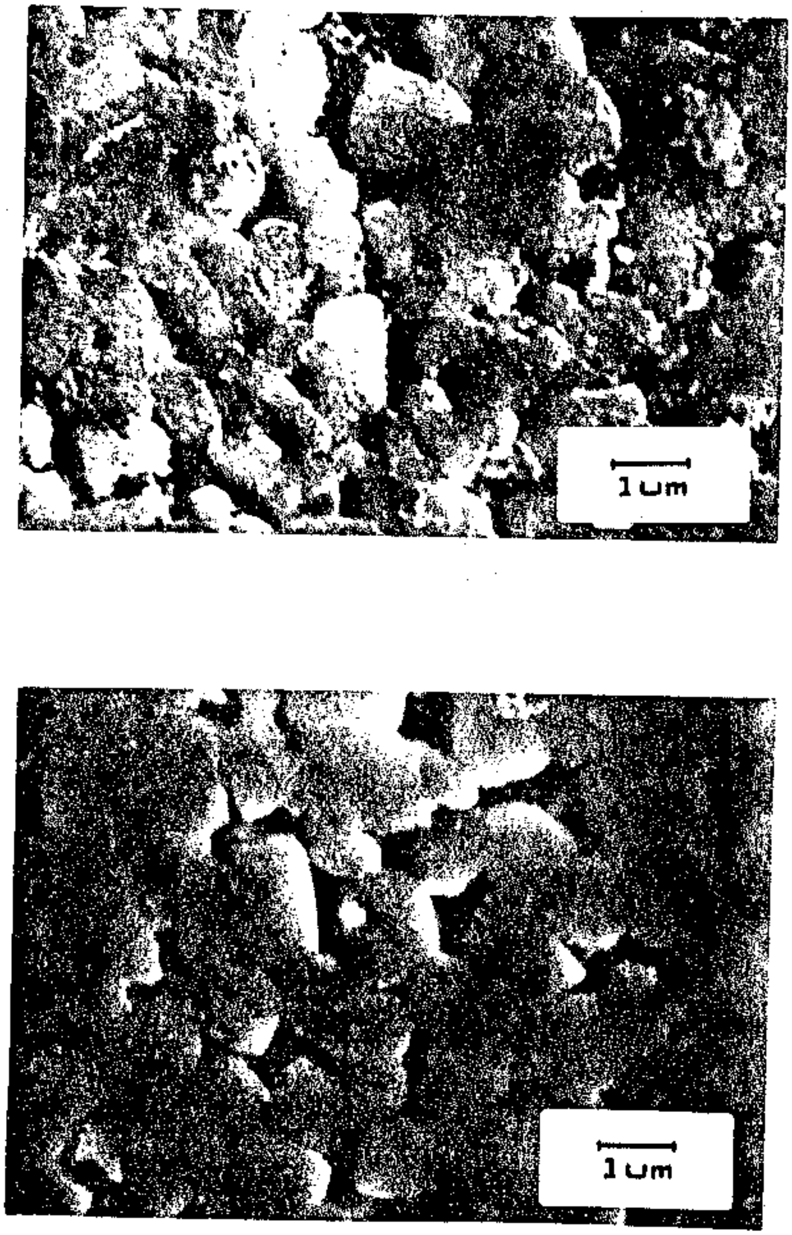


Fig. 5 - Typical microstructures (deeply etched) of HIPed specimens made from:

a) Ferrotitanium A;
b) Ferrotitanium B.

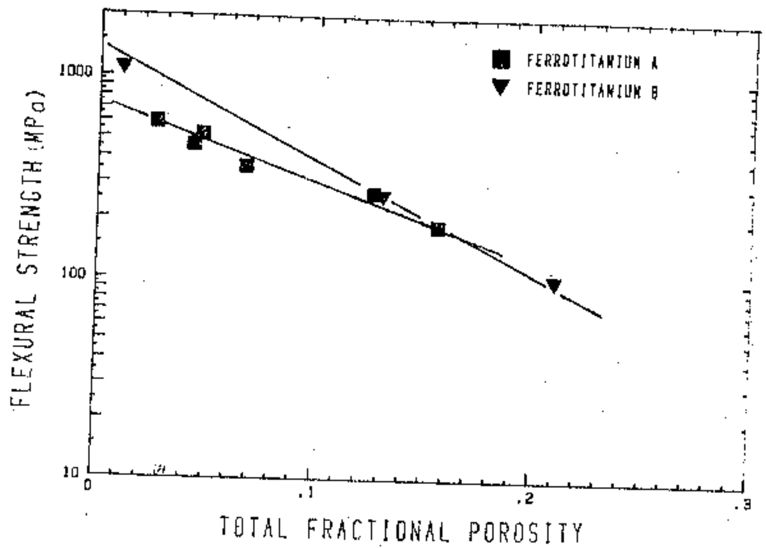


Fig. 6 - Influence of total fractional porosity on the flexural strength of HIPed parts.

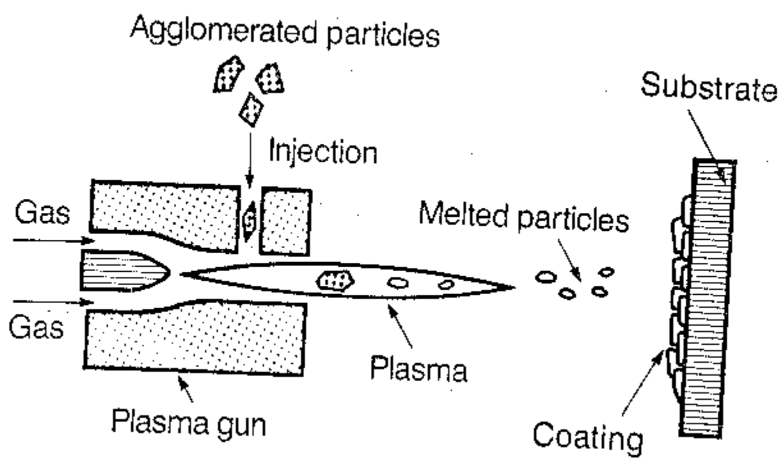


Fig. 7- Schematic view of the "Plasma Spray Synthesis" PSS process.

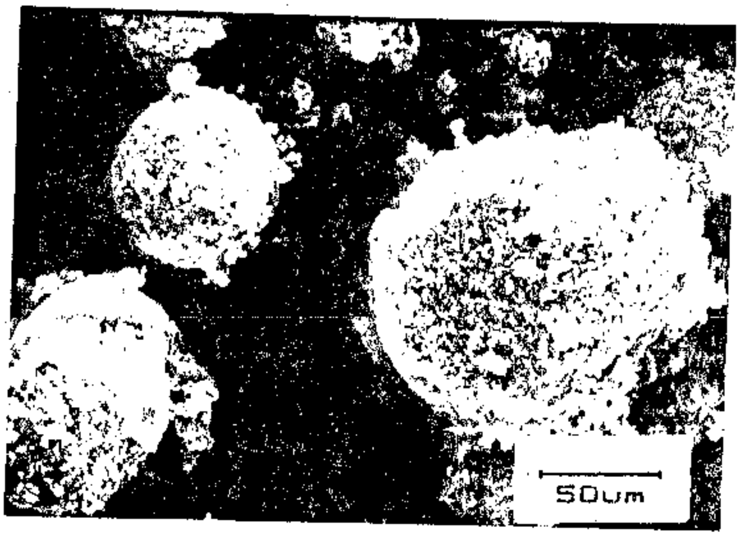


Fig. 8 - Typical appearance of micropellets before spraying.

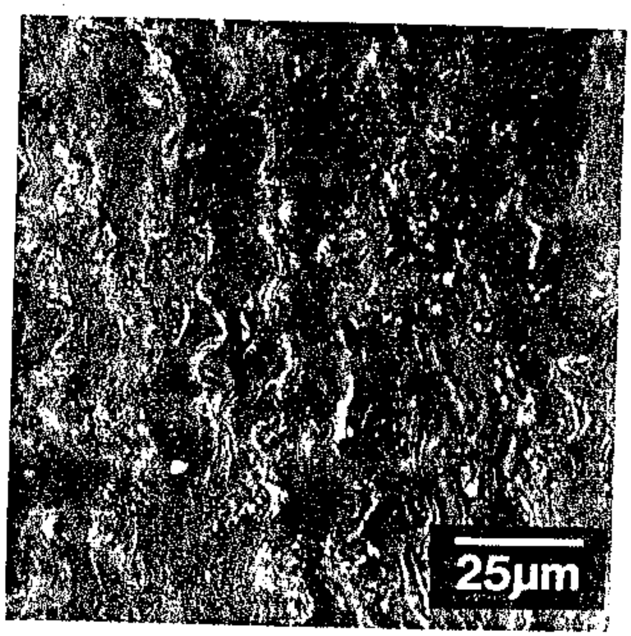


Fig. 9 - Microstructure of a SHS TiB_2-Fe coating.