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# Plasma spray consolidation of materials

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## ABSTRACT

Plasma spray deposition, long considered a coating process for applying thin protective layers has experienced recent major advances. Technology, such as the introduction the process into vacuum, the development of higher enthalpy plasma torches and the fabrication of composite materials deposition, as well as others has enabled a wide range of structural applications for the plasma spray deposition process. Spray forming of free standing shapes of ceramics, metals, intermetallics, and composite materials,with or without fibers, for producing engineered microstructures for advanced applications is now accepted as feasible and a viable plasma deposition process.

Droplet consolidation, the result of spray forming, at the substrate is a critical stage in the overall processing approach which influences a materials microstructure. This in turn affects the property evolution and performance of the material system being studied. Plasma spray consolidation and the resultant microstructure/property evolution is reviewed for nickel base alloys, refractory metal alloys, and particulate reinforced materials; each having their own characteristic microstructure. The processing-structure- properties discussion focuses on key processing factors. The promise and pitfalls of this type of forming are presented in a manner to provide the basis for future research in plasma spray forming.

## INTRODUCTION

Plasma spray forming is an emerging process technology for producing near-net shaped materials. Research conducted by NASA and others [1,2] have shown that the plasma spray coating process produces metallurgically sound, rapidly solidified deposits of many different compositions or combination of compositions [3]. The ability to process particulate materials (i.e. metal powders, discontinuous fibers, etc.) makes plasma deposition a viable processing method to produce and manufacture most of the new generation of advanced materials (i.e. rapidly solidified powders, aluminides, carbon fibers) which are being considered as engineered composites for aerospace structural applications. The overlay nature of deposition also adds substrate flexibility to the process. In the production of metal matrix composites(MMC), the metal component is "delivered at the substrate as molten metal particles, independent of the plasma jet. In turn, fibers are introduced at the deposit surface independent of the melting particulates. Plasma spray deposition, a mature "coating" process, has wide application; however, its application as a high volume manufacturing technology for making specialty materials is new.

Historically, plasma spray deposition has been used for coatings for corrosion and wear protection, thermal insulation or repair. However, recent process developments in controlled atmosphere deposition has enabled this technology to be used for the production of powder metallurgy (P/M) structures, composite metal structures, metal/ceramic matrix materials, superconducting oxide processing, and near net shape manufacturing[4]. Metals, ceramics, composites of metals and/or metals and ceramics have now been deposited using a wide range of processing equipment . The deposit thicknesses, typically for coatings (0.1 -1 mm) have more recently surpassed 25 mm for structural deposits, including the introduction of continuous or discontinuous reinforcing phases. Structural deposition applications require specific microstructure or chemistry for deposit performance. Hence, the understanding and control of microstructure evolution during plasma spray deposition is of paramount importance for the widespread use of this technology. In this paper we address these issues.

## BACKGROUND

There is no doubt that plasma processing and synthesis of materials is a multi-disciplinary activity. In this vein, the background and introduction of the lexicon used in materials science is briefly reviewed below.

### Rapid solidification

Rapid solidification is a term applied to a family of processes which, through high cooling rates, typically $> 10^4$°C, prevent diffusion related equilibrium to be established, thus, limiting the partitioning of elements and the nucleation and growth of grains. These effects lead to i)fine grain sizes (<1μm), ii) reduced material texture, iii) finely dispersed phases, iv) chemical homogeneity, and v) extended solubilities of the elements. Figure 1 shows the difference in microstructural uniformity achieved between a conventional casting of Rene 80, a nickel based superalloy, and the equivalent alloy made from fine, rapidly solidified powders (particulates). Note how the etchant highlights segregation of elements over a much larger scale in the cast Rene 80. The material properties of the more uniform P/M Rene 80 is more controllable and leads to improved performance. Materials processes, other than P/M, which have also been used to produce rapidly solidified materials include strip casting, melt spinning, laser or e.b. glazing, droplet quenching, or gas atomization. All of these processes depend on creation of a high surface to volume ratio for the cooling liquid as it transforms to solid, as well as an increased heat transfer coefficient during the liquid to solid transformation. The rapidly solidified particulates, strips and/or particulates after quenching subsequently require a post consolidation step to create useable engineering structures. Droplet consolidation/spray forming is one technique which achieves sufficiently high cooling rates yielding the benefits of rapid solidification, provided the latent heats of fusion and/or other process heat is extracted to alleviate post deposition diffusion effects.

### Particulate processing

Powder metallurgy, ceramics, discontinuous composites, spray casting and plasma spray deposition all have particulate processing in common. These processes all utilize particulates during part of their cycles. High surface to volume ratios are a characteristic of particulates, whose constituent sizes range from <1 µm to 200µm. Typically particulates have been produced from a liquid or have been precipitated from a gas and have rapidly solidified structures. The production of engineering structures from these particulate materials can involve liquid, if cooling rates are rapid, and solid state transformations making the particulates into a consolidated structure. Figure 2 is a graph showing how particulate processing (superalloys and ceramics) has been a major method of improving the materials' temperature capabilities in jet engine components. Particulate processing has also been utilized as a method of creating composite property structures through graded structures, particulate reinforcing, or fiber infiltration.

### Plasma process development

Plasma spray deposition, both D.C. arc and R.F. discharge, is a form of particulate processing where consolidation is achieved by injecting and melting particulate materials in thermal plasma jets. D.C. arc plasma deposition consists of an electric arc confined in water cooled nozzle where selected gas(es) are passed around the arc for heating and stabilization. The gases are superheated by the plasma column of the arc and are expanded through the confinement nozzle. Particulate materials are injected into the jet and are melted and rapidly cooled on impact at the substrate. Figure 3 illustrates some typical structures of a nickel base alloy that have been spray deposited. Consolidation of the particulates is achieved through rapid quenching of droplets, where highly localized solidification events yield rapidly solidified structures. Figure 4 is a transmission electron micrograph (TEM) showing the resultant fine grain size ~ 0.25µm found in "splats" from impacted molten particles of a nickel based superalloy, Rene 80[5]. This evidence of rapid cooling rates and limited grain growth indicates the level of cooling rates in this process to be $>10^5$° C.

R.F. plasma jets have also been utilized[6] where an R.F. induction source operating between 400KHz to 4 MHz creates an alternating electromagnetic field, inducing eddy currents for heating process gases passing through the center of a quartz confinement tube. Particles are injected through the center axis of the tube and pass through the "plasma" fireball where melting occurs. Though the resultant microstructures by R.F. plasma deposition resemble those produced by the D.C. plasmas, the characteristics of the droplets upon impact, i.e., droplet velocity, and heating residence time in the plasma jet are quite different and has been found capable of melting larger particles than the existing D.C. plasma gun designs[7].

Plasma particulate processing for rapidly solidified engineered materials has largely been successful due to the development of controlled atmosphere deposition[8,9]. The atmospheres have varied from low to atmospheric pressure inert gas environments. The plasma jets are strong hydrodynamic pumps which entrain significant parts of the surrounding environment into the particulate processing/melting zones, generally causing reactions to form oxide and/or nitride contamination in the deposit. Chamber spraying has been shown to be able to reduce the entrainment of detrimental atmospheric gases, limiting deposit interstitial oxygen and nitrogen to about 10-50 ppm above the starting powder gas contents[4]. Control of deposit residual stresses and increases in deposit densities, through substrate preheat, have also aided the development of structural deposits of materials from refractory oxides to refractory metals. The control of material properties through the use of plasma jet deposition requires a fundamental knowledge of microstructural evolution in the various metal, cermet, and ceramic systems. Each system reacts differently in the thermally and chemically active environment of the plasma jet, wherein each system responds differently to the resultant rapid solidification. Microstructural evolution and property development of several materials systems consolidated from particulates by D.C. plasma arc deposition are presented below.

## MATERIALS CONSOLIDATION: PROCESSING AND RESULTS

Figure 5 schematically illustrates the vacuum plasma spray deposition system used to consolidate all the alloys reviewed below. The atmospheres, which were also the arc gases were argon/helium and/or hydrogen, dependant on the jet enthalpy level required to melt the particulates. Table 1 reviews the parameter ranges of the Electro-Plasma, Inc. EPI 03 plasma gun used to make deposits of the materials systems discussed below.

In all cases, unless otherwise stated,the materials were deposited onto preheated (~600-1100°C) steel or nickel base substrates in thicknesses from 2-12mm, although thicknesses have exceeded 50mm. Materials have been evaluated in the as deposited and in the post deposit heat treated conditions through standard metallographic

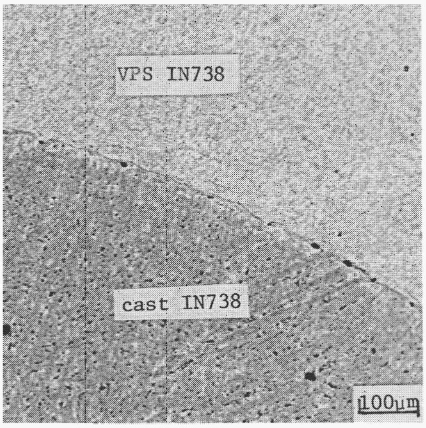


Figure 1. Cast vs. vacuum plasma deposited Rene 80 nickel based superalloy.

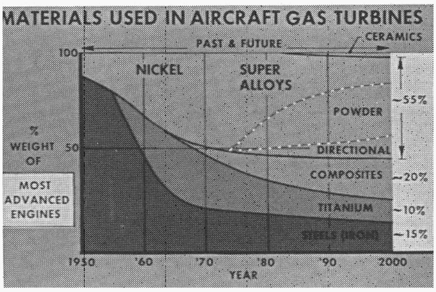


Figure 2. The chronological changes to advanced materials for aircraft engines.

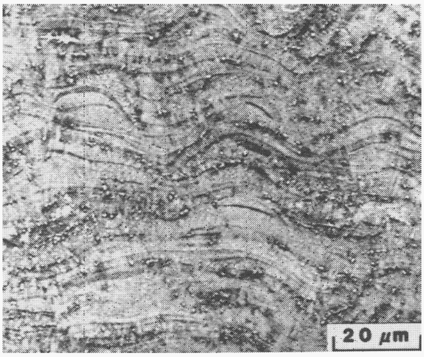


Figure 3. The as deposited microstructure of vacuum plasma sprayed Rene 80.

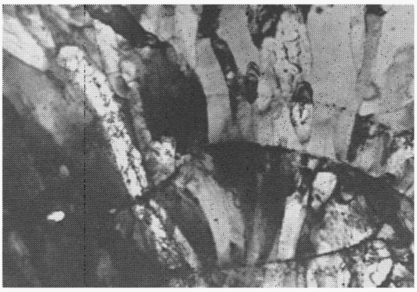


Figure 4. A TEM micrograph of the rapidly solidified structure in vacuum plasma deposited Rene 80.

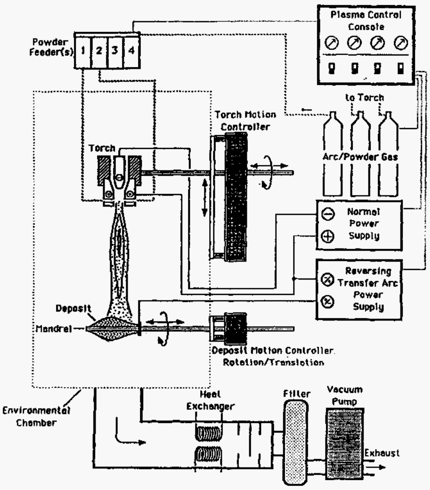


Figure 5. A schematic illustrating of the vacuum plasma spray equipment.

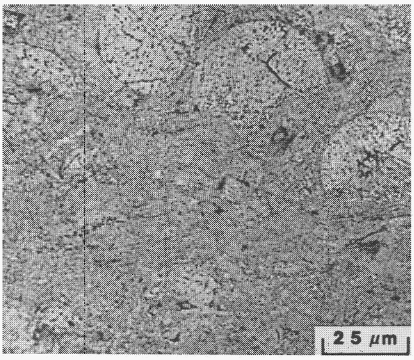


Figure. 6. A photomicrograph of vacuum plasma deposited Rene 80, illustrating partial melting.

TABLE 1. Vacuum Plasma Parameter Ranges

| Parameter | Ni- Base | Refractory Metal | Composites |
|---|---|---|---|
| Chamber Pressure | 30-100 torr | 100-300 torr | 50-200 torr |
| Gases | Ar/He | Ar/He/$H_2$ | Ar/He |
| Flow (Prim/Sec.); slm | 275-300 / 50-100 | 150-270 / 70-125 | 150-300 / 50-100 |
| Voltage; volts | 45-60 | 45-60 | 45-60 |
| Current; amps | 1200-1600 | 1100-2000 | 1100-1600 |
| Nozzle design | EPI -80, 93,111,114 | EPI -70,-93,-111 | EPI -80,-93 |
| Post Deposit Heat Treat | 1000-1150°C | 1200-1650°C | 1000-1650°C |

TABLE 2. Alloy Compositions

| Alloy | Alloy Type | Nominal composition(wt%) |
|---|---|---|
| Rene 80 | cast superalloy | Ni-9.5Co-.016C-14Cr-4.0Mo-4.0W-3.0Al-5.0Ti-.0015B-.03Zr |
| U-700 | wrought superalloy | Ni-14.5C0-0.07C-14.6Cr-4.2Mo-4.3Al-3.35Ti-.0015B |
| C103 | wrought refractory metal | Nb-10Hf-1Ti |

techniques, TEM, and chemical/gas analysis. Table 2 sumarizes the compositions of the alloys deposited in this study.

## Nickel base alloys

Figures 6 and 7 show the as deposited and the post deposit heat treated structures of Rene 80, a nickel based superalloy. The material is > 99% dense in both conditions, however, the microstructures vary as the degree of particulate melting changes. Note, in Figure 6, the circular structure of the particulates which retain significant parts of the original powder injected into the plasma jet. Carbides from the Rene 80 composition are aligned along splat interfaces and partially, fragmented particles can be seen. Figure 7 shows how after heat treatment these "unmelted" particles are retained and pin the adjacent grains' growth. This pinning was particular to this alloy. Grain sizes in the as deposited Rene 80 range from 0.25μm to 50μm. The more melted structure, Figure 3, lead to a more uniform heat treated structure and a larger grain size. A "leaner" (low alloying content) nickel base alloy, U-700, with a low carbide content is shown in Figure 8, in the post deposit heat treated condition. The figure shows an entirely different, fully recrystallized structure. This further affirms the plasma depositions' particles by the plasma jet can eliminate any of these affects. Deposit grain size and texture are controlled by substrate heat transfer from the plasma jet and to the substrate itself. Deposition must consider plasma temperature, plasma jet flow, particle trajectories, plasma/particle interaction, substrate temperatures and particle/substrate interactions.

A view from the substrate sees the arrival of a distribution of states of droplets/particles. Three major categories of particle impact are: a) solid particles plastically deform or fracture b) mushy particles (S+L) impact and deform and / or c) fully melted impact and flow as a liquid splat. Full deposit density statistically requires that the entire particle distribution impacting a deposit's surface be > 50% liquid. This provides sufficient liquid to fill and eliminate sites of "entrapped" porosity. Incompletely melted particles deform on impact. However, in gas atomized nickel alloys, gas atomization of the starting powder has lead to particulate micro-segregation causing many particles to locally fracture, see particle fragments in Figure 6, leaving voids which might not be filled by liquid from an adjacent particle impact. Refractory metal powders, generally made from crushed bulk alloys are not susceptible to this type of porosity, see Figure 11 where unmelted particle are solid and unfragmented.. However, the higher content of unmelted particles in refractory metal deposits, lead to porosity caused by particle shadowing.

Particle microstructure in nickel base alloys plays an important role for incompletely melted particles. The "mushy" impact was found to be a common occurrence in nickel base alloy deposits, allowing for particle deformation and consolidation. The large melting ranges of nickel base alloys lead to more mushy impact structures, producing high deposit densities. Refractory metal deposits did not show any mushy impacts, due to their higher, more narrow melting ranges. Hence, a particle deformation mechanism for achieving higher deposit density was not available for the refractory metal deposits.

Fully melted particles spread laterally on impact where local solidification rates are determined by "splat" thickness and surface irregularities from unmelted or partially melted particles which provide a quenching "substrate" for these molten particles. The amount and distribution of fully melted particle impact and the subsequent flow of their particle (droplets) will determine deposit densities. Deposited grain sizes have been found to be 0.25μm - 5μm for these liquid splats. However, continuous heat input from the plasma process has been shown to increase deposit grain sizes as well as to align the grains, see the TEM structure in Figure 4 [10]. Heating from the plasma jet during deposition may also anneal the stresses from the deposit as well as improve deposit cohesion through inter "splat" diffusion. Nickel base alloys, being deposited at higher homologous temperatures ($T/T_m$) exhibit more diffusion controlled transformation than refractory metals. However, the refractory metal deposits were also sensitive to the concomitant heating from the plasma jet.

Low alloy content materials with no carbides or oxides were found to grow grains during deposition obtaining a texture in the direction of heat flow, see Figure 9. Complex alloys and refractory metals were found to retain fine grain sizes (>0.25mm) due to the rapid quench rates and slow or impeded grain growth. In other deposits , not shown here, porosity was also found to retard grain growth, especially if deposit porosity levels exceeded 5%. Deposit performance depended on grain size and orientation, porosity level, phase content and the structure of reinforcing phases (i.e. carbides or oxides).

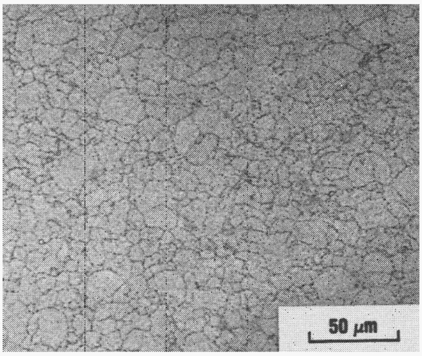
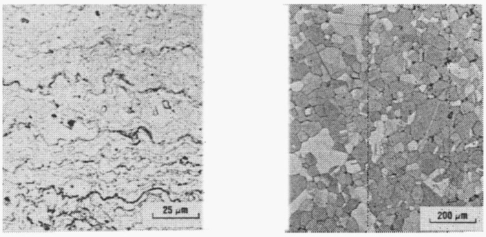


Figure 7. A photomicrograph of vacuum plasma deposited Rene 80, after heat treatment at 1120°C / 2hrs.



**As - Deposited**
**Vacuum Plasma Spray**

**Post Deposit Heat Treated**

Figure 8. The microstructure of vacuum plasma deposited U-700.
a) As deposited.
b) After heat treatment at 1120°C / 2hrs.

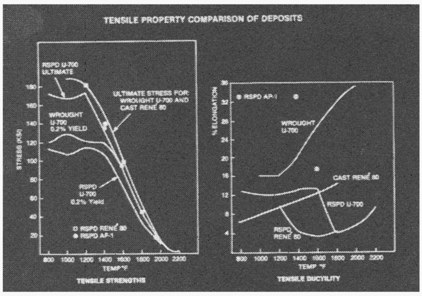


Figure 9.Tensile property comparisons of cast and plasma deposited Rene 80 and U-700 (AP-1).

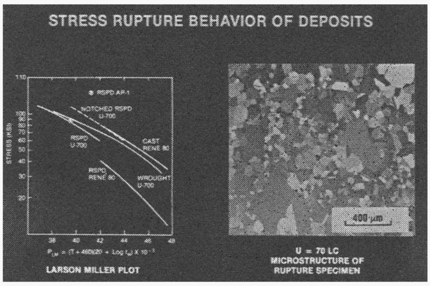
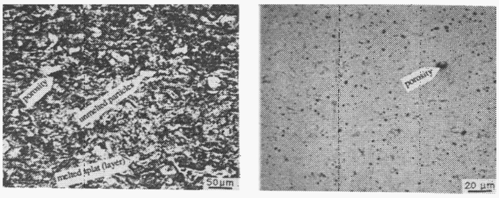


Figure 10. Stress rupture property comparisons of wrought U700 and cast Rene 80 vs plasma deposit properties.

**Niobium Alloy (Nb 10Hf 1Ti)**



**As - Deposited**
**Vacuum Plasma Spray**

**Post Deposit Heat Treated**

Figure 11. The microstructure of vacuum plasma deposited C103.
a) As deposited.
b) After heat treatment at 1650°C / 2hrs.

## Niobium Alloy (Nb 10Hf 1Ti)

### Tensile Strength Comparison

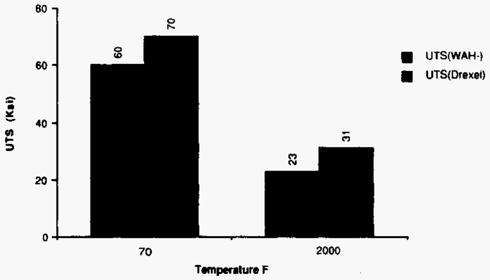


Figure 12. Tensile strength comparison of arc melted/wrought vs plasma deposited C103.

## PROCESSING ISSUES: OBSERVATIONS AND IMPLICATIONS

### Chemistry control . . . effects

The starting particulate materials for the plasma deposition process have been found to have a major effect on the post deposit and/or heat treated materials' properties. Although rapidly quenched, the compositional variations in gas atomized nickel alloy powders have been found to influence deposit density as powders fragment on rapid heating in the plasma jet. Subsequent nucleation and growth occur from the mixed structures of unmelted particle fragments, with larger grain size, to the finer grain size initiated by the 0.25μm grains nucleated from the fully molten splats. Refractory metals, produced from a more compositionally uniform wrought product and due to its higher melting range, did not exhibit fragmentation, but also had a bimodal grain size attributed to the higher content of unmelted particles imbedded in a smaller volume of material quenched from the liquid. Compositionally, however, the plasma deposits of both the nickel base alloys and refractory metals are more uniform than with cast and/or wrought methods, especially for highly alloyed or difficult to process materials and for composite materials.

process alloy flexibility; but, *the material system sensitivity. Therefore, process cannot independently* be *selected* without consideration of the effects of the material system, based on these strong interrelationships.

Figures 9 and 10 show the effect plasma spray consolidation has had on important engineering properties. The finer grain sizes have lead to tensile strengths equivalent to the cast and / or wrought version of the material. However, the ductilities and creep rupture of these materials is reduced. The deposited materials' stable, fine grain size and the increased oxygen levels (200-500ppm) have contributed to these reduced properties. Note the U700 alloy had better creep rupture properties relative to its wrought version, as compare to the cast Rene 80 alloy, due to the U700 deposits recrystallization behavior, illustrated in Figure 10.

### Refractory metals

Figures 11 shows the as deposited microstructure a refractory metal niobium alloy, C103. The density of the -44 μm niobium alloy powder deposits were >97%, lower than the previously described nickel base alloy. The porosity, as seen in the microstructure, was associated with larger (> 20 μm) unmelted particulates, as seen in the microstructure of Figure 11. The deposit structure contains unmelted particles with some degree of particle deformation, but not as high as the particle deformation seen in the nickel based superalloy deposits. In general, all refractory metal deposits showed lower densities, lower as deposited splat interdiffusion, and less recrystallization, than the corresponding nickel based alloys. This has been attributed to the lower homologous plasma processing temperatures ($T/T_m$) of the refractory metals. These refractory metal deposits also show less post "splat" diffusion and heat treatment recrystallization than the nickel base alloys under similar deposition parameters. Thus, the lamellar nature of the refractory metal deposits is more apparent, even after heat treatments as high as 1150°C..

Figure 12 illustrates the relative strength of the plasma spray consolidated niobium alloy (C103) to commercially available conventional P/M processed and wrought condition. Note the increased strength of the plasma deposited material, which had been post deposit heat treated at 1650°C prior to testing. The increased strength is attributed to the finer grain size and the higher oxygen (1500 ppm vs <100 ppm for wrought materials) content of the plasma consolidated materials. The higher oxygen content originated from the powders and is also responsible for the low ductilities of the plasma consolidated material, 1-5% Elongation vs. 20% for the wrought materials.

### Particulate reinforcement

Particulate reinforcement of plasma sprayed deposits is a useful attribute of the process which utilizes particulate materials as raw material. A matrix with a strong second phase is typically used in wear applications or for improving the structural strength of materials. Particulates have been introduced both as mechanical mixtures or by pre-agglomerating a powder mixture of the matrix and the second phase. Figure 13 is a photomicrograph of a air plasma deposited iron base alloy/45 vol% TiC composite plasma deposit with ~ 3-5 μm TiC particles introduced from pre-agglomerated (spray dried), and densified powders. Note the high content of TiC particles in the structure and a retention of the TiC particle shape. Correspondingly, a plasma deposit of a composite niobium alloy (C103) matrix with 45 vol% TiC particulate loading, produced from a mechanical blend of -44μm C103 alloy powder mixed with ~3-5 μm TiC particulates are shown in Figure 14. Figure 14 shows a segregation of TiC particles to lamellae interfaces with melted (lamellae) and unmelted particles of the metal matrix . A less uniform distribution of the TiC was produced via the mechanical blend route, as determined by the lamellae spacing.

The second phase particle in both types of deposits has affected the resultant microstructure by i) pinning the grain boundaries, ii) introducing a source of porosity adjacent to second phase carbide particles, and iii) causing matrix/second phase compositional changes due to the interdiffusion of elements during the impacting and cooling of the droplets of these materials. Many of these characteristics will affect the final properties of the plasma consolidated materials.

## MICROSTRUCTURE AND PROPERTY EVOLUTION

The resultant properties of any plasma spray consolidated material is ultimately determined by the microstructure of the deposit. The evolution of deposit microstructure involves two stages, the melting and the subsequent consolidation events(deformation, cooling rate, heating time) at the substrate. Porosity, resulting from incompletely melted impacting particles reduce deposit strength and ductility. Rapid solidification, determined by

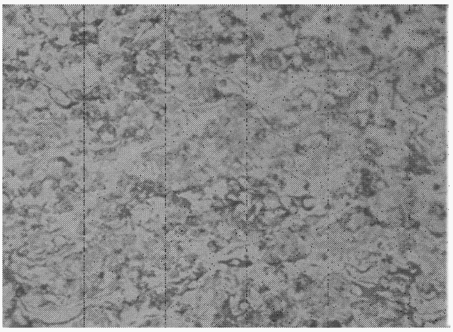


Figure 13. The microstructure of vacuum plasma deposited TiC particle reinforced steel.

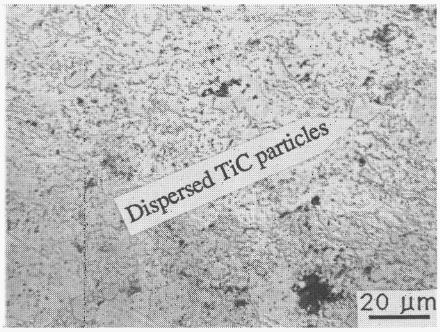


Figure 14. The microstructure of vacuum plasma deposited C103 with 45 vol% TiC particles.

the cooling rates of melted particles, suppress phases and reduce grain size; however, heating of the deposited

The one serious compositional limitation is due primarily to the purity of the starting particulates. These particulates contain an enormous surface area, especially for particle sizes < 20µm. This surface area contains thin oxide scales which in many cases cannot be easily reduced and/or retain adsorbed gases. These scales and gases are entrapped during atomization processing, creating high interstitial contamination (>1,000 ppm) which reduces the ductility and the dynamic properties of materials that are consolidated from these powders by plasma spray methods. The reduction of these gas contents in the deposits are an area of intensive research for structural plasma spray applications, many of which are sensitive to the poor ductility values produced in many of these deposits. Research is being conducted on decreasing the surface area of the starting materials through larger powder size melting capability processes, such as R.F. plasma deposition, which produces longer particle dwell times in the the plasma jet.[11]

## Limitations/processing improvements

The cost of raw particulate starting materials and deposition yields hinder the growth of applications. The yields of acceptable particulates starting sizes (~5-100µm) as well as the means of handling to reduce contamination bring the particulate materials costs over $100 U.S per kilogram. Deposition utilization of ~30% are typical and further increase the cost of the process. The cost of deposition becomes small compared to these raw materials costs; however, the specialized nature of some environments, necessary to control the proper materials structure, is another factor in the final process costs.

The management of thermal effects in the plasma deposition process needs to be accomplished through, accurate and reliable sensors and effective control means. Thermal effects can be caused by the heat load to the deposit from the plasma jet or the heats of fusion of the solidifying droplets. Thermal shocking, thermal expansion mismatch or other thermally induced stresses during plasma deposition are sources of material cracking or degradation. The source of these effects and methods to minimize them must be incorporated into any production consolidation system.

Finally, the production of composites, either continuous fibers or discontinuous fiber or particulates requires plasma processing conditions which do not significantly alter the properties of the fiber or the second phase. The integrity and uniformity of the reinforcing phase needs to be maintained through the plasma deposition process. This challenge requires methods to place and control fiber temperatures and/or control the state of the second phase, independent from the matrix to be deposited. Equipment and process development continues to address these issues.[12]

## CONCLUSIONS

Rapid solidification rate materials processing associated with P/M consolidation has been accomplished through plasma spray forming and is an alternative processing route for materials consolidation with high solidification rates, yielding near net shape, homogeneous microstructures. Deposit densities of >99% have been achieved for nickel base alloys, refractory metal alloys and particulate reinforced composite materials of these matrix bases. Plasma spray deposition spray forming offers opportunity to both consolidate powders into a composite structure, and rapidly solidifying the droplets during consolidation. The development of final deposit properties has been closely linked with plasma deposition parameters as well as the starting materials, their structure, and their properties. A major limitation of this approach to date has been the interstitial gas content ($O_2$, $N_2$, etc..) of deposits originating from the surface of the powder particles and the process economics. The plasma deposition process has been found to be compatible with fiber processing to form fiber reinforced composite materials. Droplet consolidation, inherent to the plasma process, permits thin metal matrix overlays to be deposited onto fiber structures and subsequent HIP (hot isostatic pressing) processing consolidates the fibers and the matrix. The capabilities and flexibility of the plasma process to produce near net shaped components of advanced materials are abundant and growing. Processing related materials properties and the improvement of process economics are the major challenges ahead.

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