Proceedings of the 8th National Thermal Spray Conference, 11-15 September 1995, Houston, Texas

Continuous Spray Forming of Functionally Gradient Materials

T. N. McKechnie, E. H. Richardson
Plasma Processes Inc.
Huntsville, Alabama

R. D. Watson
Sandia National Laboratories
Albuquerque, New Mexico

Abstract

Researchers at Plasma Processes Inc. have produced a Functional Gradient Material (FGM) through advanced vacuum plasma spray processing for high heat flux applications. Outlined in this paper are the manufacturing methods used to develop a four component functional gradient material of copper, tungsten, boron, and boron nitride. The FGM was formed with continuous gradients and integral cooling channels eliminating bondlines and providing direct heat transfer from the high temperature exposed surface to a cooling medium. Metallurgical and X-ray diffraction analyses of the materials formed through innovative VPS processing are also presented. Applications for this functional gradient structural material range from fusion reactor plasma facing components to missile nose cones to boilers.

A FUNCTIONALLY GRADIENT material (FGM) is a composite of at least two constituents that smoothly transitions from 100% of constituent A to 100% of constituent B through controlled gradients. A functional gradient may be several lamellae or it may be a "continuous" change in composition[1].

Early FGM research focused on high heat flux components for aerospace applications[2]. Current designs of high heat flux components employ multiple materials requiring complex bonding techniques that are troublesome to manufacture and can be operationally sensitive. A gradient material can be used to optimize the design of the high heat flux component. Advantages of incorporating a FGM include:

- minimizes differences in thermal expansion between different materials which reduces thermal stresses
- eliminates complex bonding and fabrication problems
- eliminates the reduction in heat flow through dissimilar material bond joints
- non-destructive examination by techniques such as ultrasonic inspection of bond joints is made simpler with the continuous gradient structure
- optimizes design by placing the best materials in the best locations
- can be made to near net shape

The objective of this project was to vacuum plasma spray (VPS) a material for use as a plasma facing component in next generation fusion reactors including the International Thermonuclear Experimental Reactor (ITER). Among the many material properties required for plasma facing components are low plasma poisoning potential (low atomic number), good high temperature mechanical properties, and high thermal conductivity. Due to these various properties, a functionally gradient material was proposed to meet all the material requirements.

Technical Approach

The Gradients. The gradient material designed by the authors for use as a plasma facing component started within a high conductivity material (copper) to optimize heat transfer to the coolant and graded to the main wall material (high strength tungsten). Next, the component graded to a low atomic number surface material (boron) to limit plasma poisoning potential. Finally, the surface of the low atomic number material was nitrided (to form boron nitride) to make it inert to most fabrication, handling, and environmental problems.

Integral Cooling Passages. Thermally efficient cooling passages were fabricated within the functional gradient plasma facing component structure. These were designed to allow maximum heat transfer from the plasma facing surface to the cooling medium. The design would

This work was supported by the United States Department of Energy under Contract DE-AC04-94AL85000.

increase heat dissipation because no bondline interruptions would exist between the coolant and plasma surface. Present designs feature complex bonding techniques which can only provide near continuous contact between materials. The incorporation of active cooling channels within the gradient eliminates the possibility of catastrophic failure at bondlines.

The design for the cooling passages had to account for two opposing factors. First, from a heat transfer standpoint, high aspect ratio cooling channels have shown to improve heat transfer to the coolant. NASA Lewis testing[3] has shown a 30% reduction in hot wall temperatures in rocket engines when the coolant passages were increased from an aspect ratio (ratio of channel height to width) of 0.75 to 5. From a manufacturing standpoint, the higher the aspect ratio becomes the more difficult it becomes to vacuum plasma spray form[4]. As channels become narrower, VPS material tends to adhere to the sidewalls prior to complete channel fill so that a bridging of material takes place. This phenomenon can be overcome to some extent by designing the cooling channels to be more triangular (i.e. the hot side channel width is narrower than the cold side) than rectangular. Based on this research, three channel configurations were designed and fabricated:

Table 1: Configurations of integral cooling channels

Channel Width Hot Side	Channel Width Cold Side	Channel Angle	Depth	Channel Location
0.020 in.	0.040 in.	45 degrees	0.035 in.	FGM
0.020 in.	0.040 in.	45 degrees	0.075 in.	FGM
0.060 in.	0.060 in.	90 degrees	0.150 in.	Cu cooling panel

Thermal Spray Process. The Vacuum Plasma Spray (VPS) process was used to fabricate the gradient materials. VPS offers the ability to fabricate functionally gradient structures through a repeatable, computer-controlled process. Multiple powder feeds into the torch were used to deposit the different materials and gradients. Other advantages of the VPS process include the ability to fabricate materials in a non-oxidizing environment critical for material properties.

The combination of correct materials, proper design, and VPS generated a high heat flux, plasma facing component material with the following operational advantages:

- no plasma poisoning from high atomic number materials
- no bondlines between plasma facing surfaces and coolant channels
- high thermal efficiency for heat dissipation between the surface and coolant
- repairable through plasma spray techniques
- inexpensive and commercially viable

Technical Procedures

The procedures used in fabricating the high heat flux component with integral cooling channels is outlined below (also see Figure 1):

1. Steel and graphite mandrels were machined with integral cooling profiles.
2. The functionally gradient Cu/W/B/BN material was deposited onto the mandrel.
3. The mandrel was removed by mechanical means.
4. A copper back plate was brazed to the tips of the cooling channel fins to form the channel closeouts.

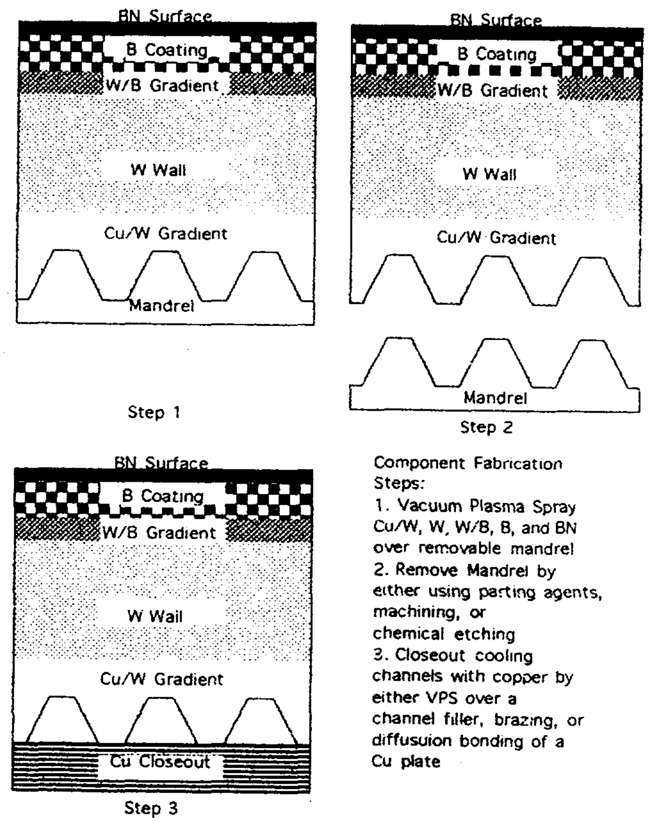


Figure 1: Fabrication sequence for functionally gradient high heat flux components with integral cooling channels

Vacuum Plasma Spray of Copper/Tungsten Functionally Gradient Structures. The main obstacle faced in the production of functionally gradient structures was choosing a plasma spray gun anode/nozzle which could spray all powders consecutively. The author's standard parameters call for different anodes for spraying copper and tungsten (spraying of boron was completely new). The EPI 03CA-93 anode was selected for all powder parameter development based on its known ability to spray copper and tungsten.

The authors evaluated parameters for depositing -325 mesh copper and tungsten powders using Drexel

University's VPS equipment. The authors had, in the past, developed these parameters for other VPS systems, but adjustments had to be made in spraying these powders at Drexel due to differences in equipment. Steel coupons were sprayed individually with copper and tungsten for deposition efficiency calculations and microstructural evaluations. The sprayed samples were then compared with previously characterized samples.

Once parameters were optimized for the Drexel system, continuous gradient structures were fabricated on steel and graphite plates. Variations in powder feed rates and carrier gas flows were used to control the gradients. Each sample was cross-sectioned and metallography was performed.

Vacuum Plasma Spray of Boron. Boron agglomerated to -140/+270 mesh size was used for plasma spraying parametric development. Steel and tungsten coated coupons were used for deposition studies. Once thick deposits of B were achieved on W, samples were cross sectioned and metallurgically evaluated.

Vacuum Plasma Spray of Boron/Tungsten Functional Gradient. The optimized parameters determined for spraying W and B were utilized in the forming of B/W functional gradients. Again powder feed and powder carrier gas flow were altered to achieve a continuous gradient structure. Samples were prepared and metallurgically characterized.

In-situ Nitriding of Boron Surfaces. To nitride boron, several techniques were evaluated:

- VPS of boron with a nitrogen plasma
- VPS of boron with an Ar/H_2 Plasma with N_2 powder carrier gas
- VPS of 50%B/50%BN agglomerated powder with a N_2 powder carrier gas

Preliminary Specimen Fabrication. Proof of concept (POC) specimens were fabricated with a VPS gradient structure of Cu/W/B/BN. This was achieved by using the optimized parameters developed for each powder. Once POC specimens were formed, some were cross-sectioned and metallographic analysis was performed. Other POC specimens were prepared for brazing to a copper coolant manifold. A schematic of the brazing operation is shown in Figure 2. Two POC specimens have been brazed to a copper closeout and pressure tested. One specimen was a flat plate POC and one was a channeled POC.

Research Results

Metallurgical Analysis. Metallurgical analysis was performed on all materials sprayed. Plasma spray parameters were recharacterized or developed for each material. Samples were sectioned, mounted, polished and evaluated. Metallographic concerns such as porosity, grain structure, and grain size were examined. The following text contains micrographs of the functionally gradient materials developed by the authors.

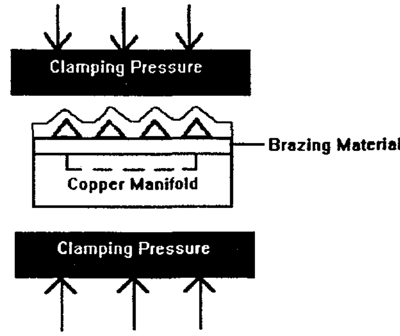


Figure 2: Schematic of Brazing Operation

The following micrograph, Figure 3, shows the full gradient from 100% Cu to 100% W and dramatic evidence of the functionally gradient structure.

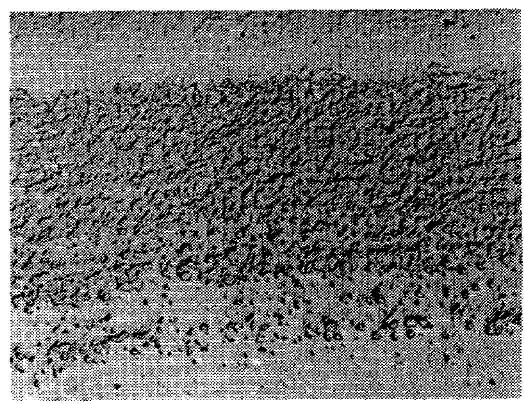


Figure 3: Copper/Tungsten functionally gradient specimen. Taken of specimen PP 93-40 at 50X, as-sprayed, unetched. Notice 99+% density and intimate bonding.

Figure 4 shows the functional gradient of three materials, i.e., from 100% Cu to 100% B with a surface of BN. X-ray analysis of B/BN deposits is presented demonstrating the success of VPS processing of BN.

Material property analysis such as tensile testing or thermal conductivity testing was not performed on the gradient materials. However, much information can be gained from an examination of an etched sample. Presented below are micrographs of etched vacuum plasma sprayed copper alloy NARloy-Z and tungsten and as-sprayed boron which have been deposited by the authors. A fully dense, recrystallized VPS microstructure is known to have mechanical and thermal properties approaching those of cast or wrought material[5].

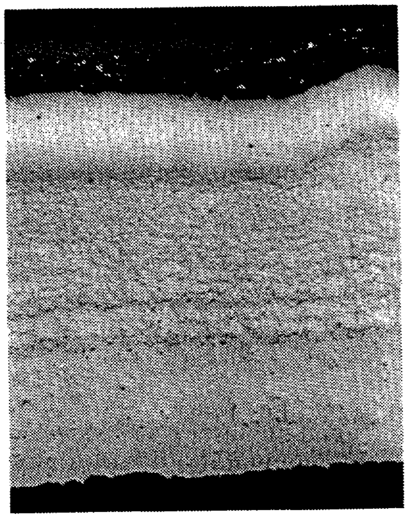


Figure 4: Copper/Tungsten/Boron/ Boron Nitride functionally gradient specimen. Taken of specimen PP 93-40 at 31.25X, as-sprayed, unetched.

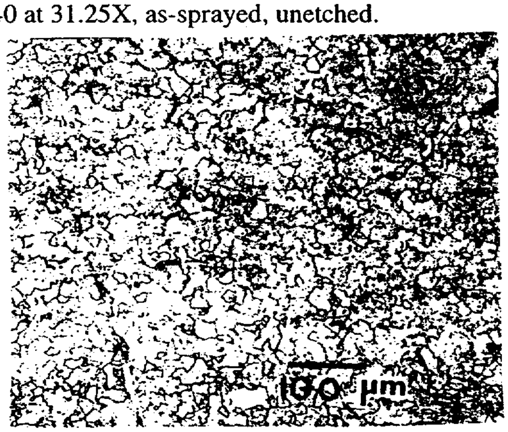


Figure 5: Dense, fully recrystallized vacuum plasma sprayed copper alloy NARloy-Z, etched, 100X

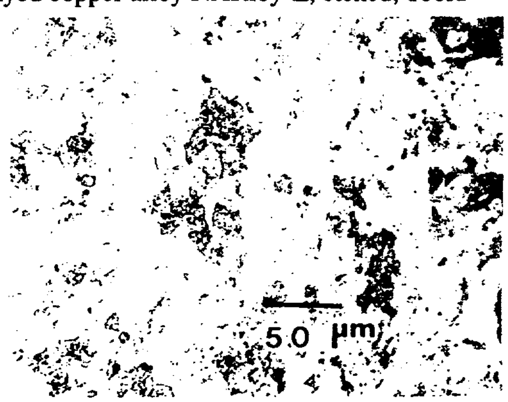


Figure 6: Dense, fully recrystallized vacuum plasma sprayed tungsten, etched, 200X

The authors also successfully sprayed samples with integral cooling channels. These specimens demonstrate proof of concept for using VPS as a method of plasma facing component manufacture. The design of these cooling channels is described earlier in this paper. In Figure 8 a cross sectioned view of the innovative, high efficiency cooling channels is presented.

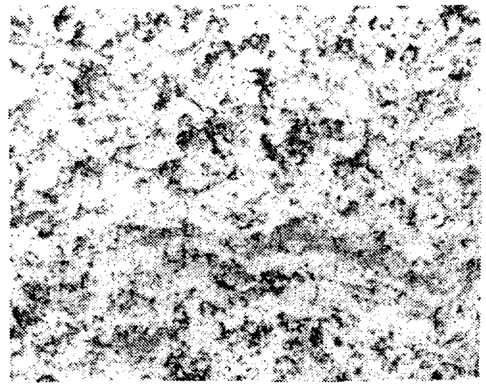
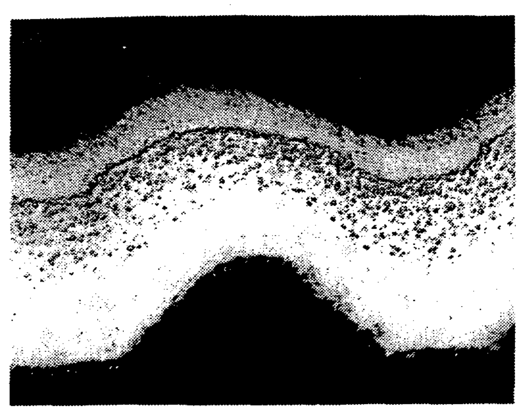


Figure 7: Dense, as-sprayed boron, 200X

8a) Cross sectioned view



8b)Cross sectioned view at 25X. Note the gradient structure

Figure 8: Proof of concept specimen with integral, high efficiency cooling channels

X-Ray Diffraction analysis of B and BN. X-Ray Diffraction analysis was performed at Drexel University to demonstrate the success of VPS processing of boron nitride powder, and other in-situ nitriding techniques mentioned earlier in this paper. A Seimens KRISTALLOFLEX with a current of 30mA and a voltage of 40kV was used in this analysis.

Figure 9 shows the X-Ray analysis of run PP93-40 (Figure 8). The analysis verifies the existence of BN on the surface. The W peak is prominent as the thickness of the low atomic number B/BN deposit is not substantial enough to block the high atomic number W transmission.

The method of in-situ nitriding that provided the best results was the third method mentioned above. This method consisted of vacuum plasma spraying an agglomerated powder of 50% boron and 50 % boron nitride. Nitrogen was used as a carrier gas.

Proof of Concept Specimen (POC) Fabrication. Cu/W/B/BN functionally gradient materials were successfully Vacuum Plasma Sprayed with and without integral cooling channels. Cu/W/B/BN FGMs were made on graphite and steel plates and subsequently removed. The spray deposits over the raised channels retained the channel configuration (Figure 8). The effect of raised channels in a plasma facing component application is unknown, but raised channels do cause difficulty in brazing. Clamping pressure on the braze joint to the copper manifold was distributed through the raised channels and not uniformly throughout POC surface. Therefore the braze joints were not intact on the POC specimens perimeter which led to water leaks during pressure testing.

A second POC specimen with cooling channels in the copper manifold did not braze correctly due to distortion in the functionally gradient material. A warpage in POC was not mitigated by high clamping pressure, leading to poor braze joints in several areas. The poor braze joints leaked water during pressure tests.

Conclusion

The author's preliminary efforts on this project have demonstrated that multi-component functionally gradient materials can be vacuum plasma spray formed with continuous gradients. Furthermore, multi-component functionally gradient materials have been vacuum plasma spray formed with integral cooling channels. These samples have been brazed to copper closeouts and pressure testing has been completed. These tests have demonstrated the soundness of the vacuum plasma sprayed FGM as failure always occurred in the braze joint and not in the FGM itself. Further improvements in the spray forming of continuous gradients will include computer controlled powder feed transition.

Acknowledgment

This work was completed under a Phase I Small Business Innovative Research (SBIR) grant from the U. S.

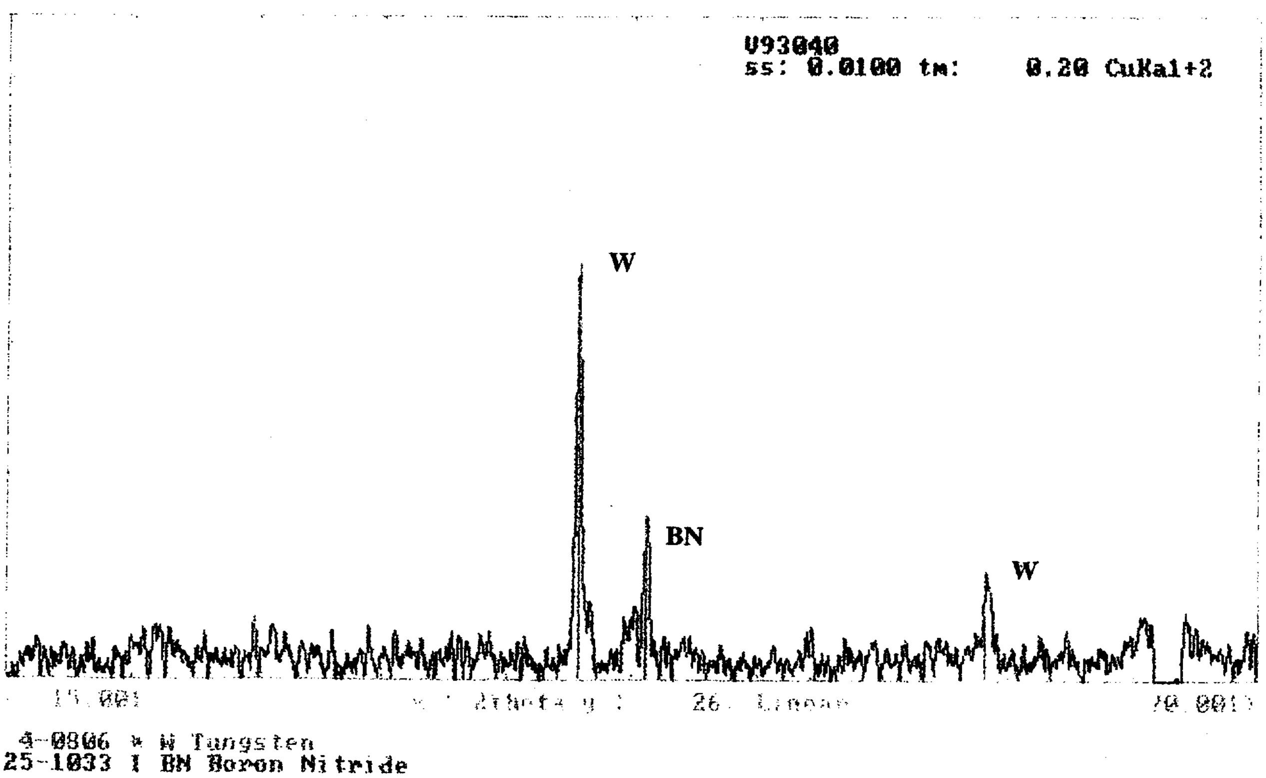


Figure 9: X-Ray diffraction analysis of specimen PP93-40

Department of Energy, grant number DE-FG05-93ER81563. Special thanks are extended to Mr. Marvin M. Cohen and Ms. Mary Lou Crow of the U.S. Department of Energy, and Dr. R. D. Watson of Sandia *National Laboratory, Albuquerque.* The authors also wish to thank Ron Smith and Don Gentner of Drexel University for their assistance.

References

1. A.M. Glaser, M.L. Shalz, B.J. Daldleish, A.P.Tomisa, " A Transient FGM Interlayer Based Approach to Joining Ceramics," Ceramic Transactions, Functionally Gradient Materials, J. Birch Holt, M. Koizumi, T. Hirai, Z. Munir, editors, The American Ceramic Society, Westerville, OH, 1993, Pp. 341-357

2. M. Koizumi, "The Concept of FGM," Ceramic Transactions, Functionally Gradient Materials, J. Birch Holt, M. Koizumi, T. Hirai, Z. Munir, editors, The American Ceramic Society, Westerville, OH, 1993, Pp. 3-10

3. J. Carlile, "An Experimental Investigation of High-Aspect Ratio Cooling Passages," NASA Technical Memorandum 105679 and AIAA paper 92-3154

4. P. Krotz, "Vacuum Plasma Spray Efforts," Rocketdyne MPR 90-0056 and NASA Materials Consortium Report, May 31, 1990

5. T.N. McKechnie, Y.K. Liaw, " Metallurgy and Properties of Plasma Spray Formed Materials," Thermal Spray: International Advances in Coatings Technology, C. Berndt, editor, ASM International, Metals Park, OH, 1992, Pp. 839-845