



PoC project: Investigation of new methods for the manufacturing of Copper-Diamond composites with tailored thermo-physical properties

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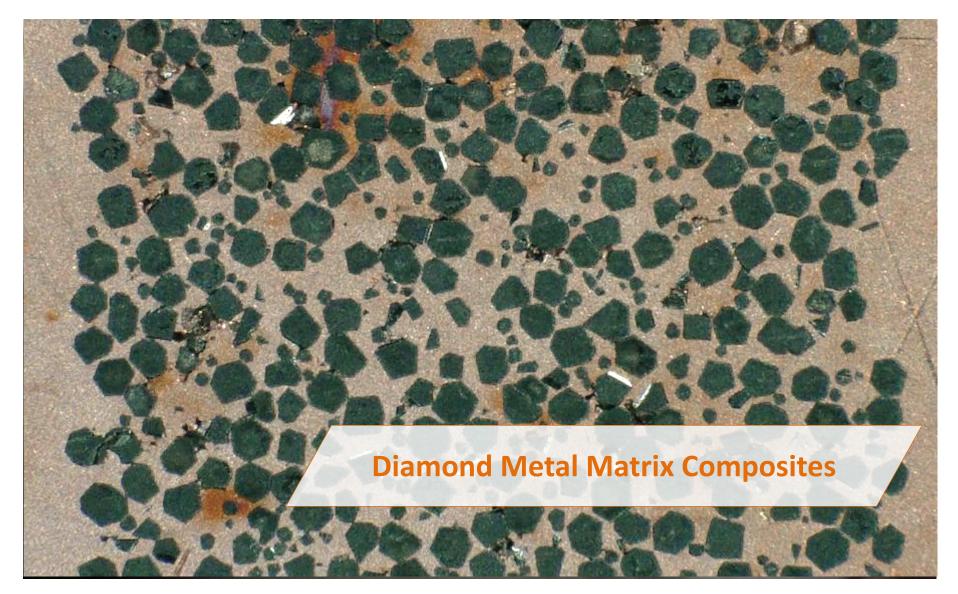
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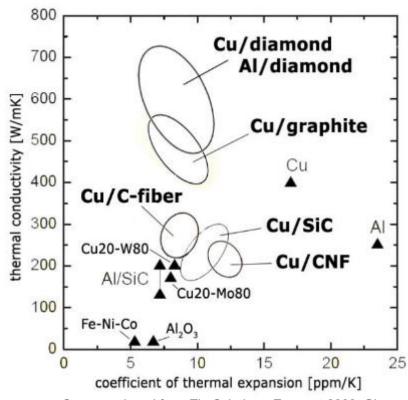








- Combining the properties of diamond and metals
- Address gap for high thermal conductivity (TC) and low Coefficient of Thermal Expansion (CTE)
- Isotropic properties
- Applications for cooling of electronics or laser components
- Potential application for collimators in LHC



Source: adaped from Th. Schubert, Euromat 2009, Glasgow



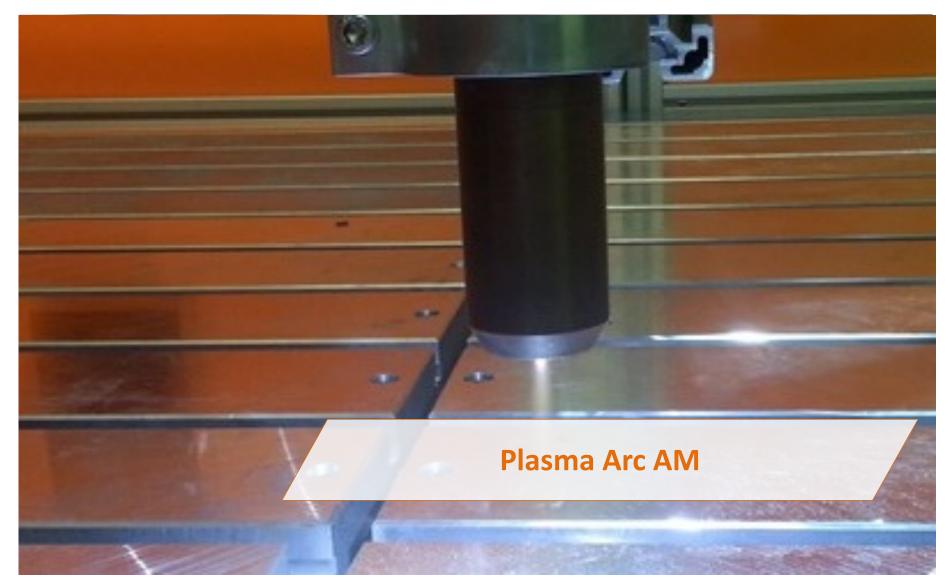


Objectives of Proof of Concept Study

- Explore the use of Plasma-Arc Additive manufacturing techniques for Diamond based metal matrix composites for building up layer by layer
 - Assessment of several manufacturing concepts to achieve samples of Diamond Metal Matrix Composites using a plasma arc process
 - ✓ Composites with >30vol% Diamond content are targeted
 - Enhanced thermal conductivity and lowered CTE due to diamond content
 - Preparation of test parts and analysis of thermal properties





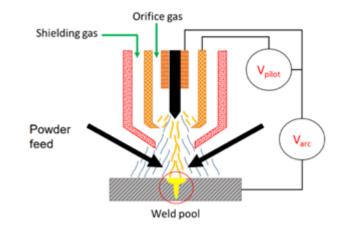




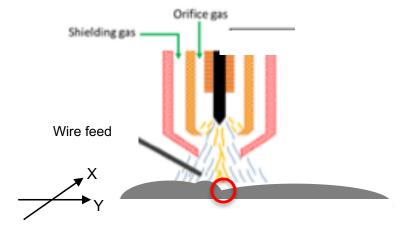
Plasma Metal Deposition (Wire Feed/Blown powder)

Blown Powder





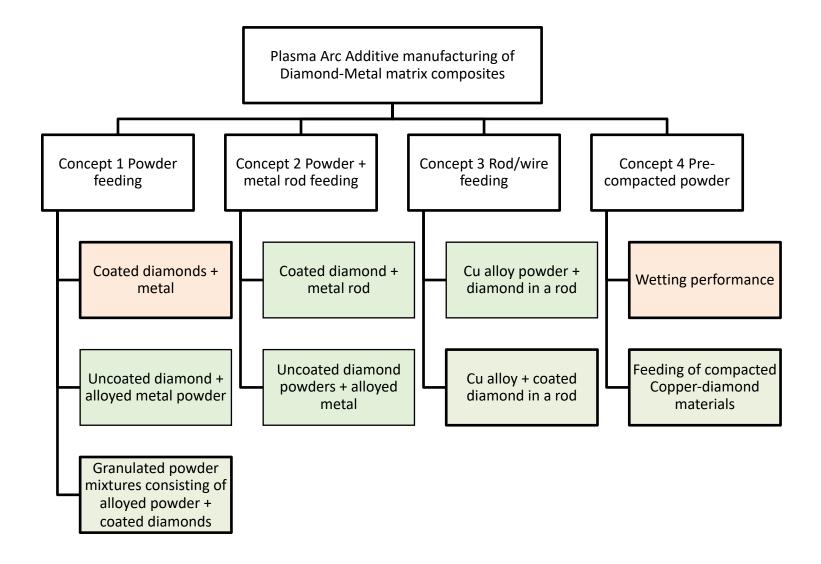
Wire Feed



Main idea: use of the Plasma Arc method for the manufacturing of metal diamond composites using blown powder/wire feed method









First assessment of using PMD process



- Issues with substrate interaction
- Diamond graphitization
- De-Wetting with the liquid melt
- Separation of the diamond
- Overspray
- Inhomogeneity
- Oxidation
- Difficult to feed using the standard powder feeder
- ..and many more
- Need for detailed analysis and separation of "challenges" into the development of smaller building blocks





Pre-cursor materials

- Challenge 1: standard powder feeding system is not suitable for processing powder mixtures containing diamonds
- Challenge 2: there are no suitable "feedstocks" (wires) available
- Challenge 3: protection the diamond/enable wetting in the matrix
- Challenge 4: Controlling/avoiding of de-wetting between diamonds and the liquid matrix
- Challenge 5: Oxygen sensitivity of the matrix





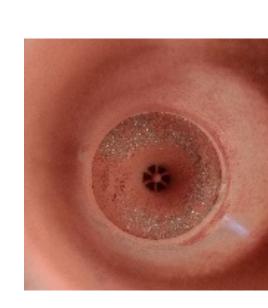
Processing of composite materials using plasma arc

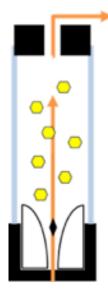
- Challenge 6: Avoid graphitization of diamond
- Challenge 7: Homogeneous distribution of diamond particles
- Challenge 8: High diamond loading (>30 vol%)
- Challenge 9: Manage homogeneity in multilayers
- Challenge 10: Achieve required thermal properties



Challenge 1: Feedstock preparation/transport for further processing

- Approach #1: Design of a powder feeding concept
- Powder feeding of dissimilar materials is difficult with standard feeding systems
 - Large diamond grains (regular)
 - Irregular metallic matrix
 - Risk of separation/segregation during feeding/clogging
 - Wear and abrasion of the moving components in classical feeding mechanisms











Blown diamond powders

- Using the feeding system a combination of Cu wire + titanium coated diamond powders was tested
- Use of Mo substrate to minimize uptake of Fe and Ni into melt pool → known graphitization catalysts
- Temperature dependent wetting of Cu on Mo unless under H₂
- H₂ dissolves in Cu at high temperatures, which then escapes during cooldown, leading to severe porosity/foaming
- Mo substrate was heating up to rapidly, leading to severe spreading of Cu
- Using of high gas flow results in severe overspray and turbulences in the powder; too less flow rate results in an oxidation of matrix.











Challenge 1: Feedstock preparation for further processing

- Approach #2: Assessment of granulation processes to enable:
 - better distribution of diamonds inside the matrix
 - Less wear and clogging issues in powder feeding system
 - Less overspray => higher gas flow possible
 - Granulates are pre-sintered



Cu alloy+ dia A19-240



Cu+diamond A19-241



Cu alloy + diamond A19-242





Challenge 1: Feedstock preparation for further processing

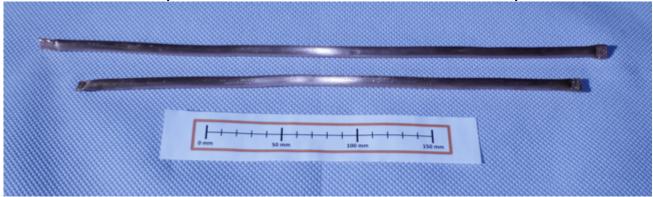
- Approach #3: Assessment of pre-compaction of plates and ingots for further processing
- Assessment of compaction/densification behavior of different alloys (Cu alloy + diamond particles) for preparation of weldable ingots
- Compaction for material preparation of up to 50 vol% of diamonds was confirmed

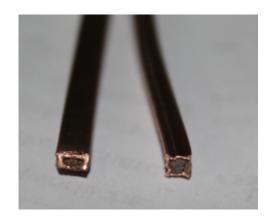




Challenge 2: No commercial filled wires available

- Approach: Demonstration of a fabrication for a metaldiamond "wire" which can be feeded into the plasma torch avoiding the severe overspray
- A process for the preparation of a filled wire was established using a Cu envelope which is filled with the metal diamond powder
- Filling of the Cu envelope can be done with a mixture of 50-60 vol% of diamond
- Including of Cu envelope a final concentration of 30-40 vol% can be achieved
- First tests were done with copper and coated diamonds; next steps are to be done with selected alloys from #4.





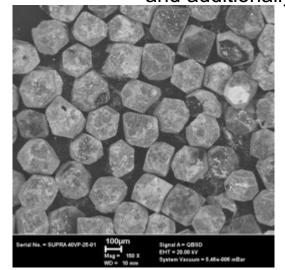


Challenge 3: protection the diamond/enable wetting in the matrix

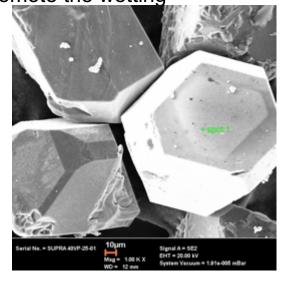
Approach: Coating of diamonds with functional layers for different diamond grain size suitable for the powder feeding

Coating was done by using thermo-diffusion processes.

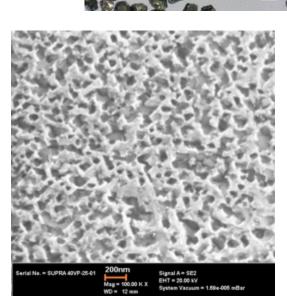
Various metals can be applied, e.g. Cr, Mo, W with different thicknesses to protect the diamond and additionally to promote the wetting



W- coating



Mo- coating



Magnification





- Combined approach: using of coated diamonds and appropriate copper alloys
- Lessons learned: only coating is not sufficient, we need both due to the short contact time
 - More than 20 combinations of Cu alloys with different coated diamonds have been assessed
 - Dewetting can be observed and result for some alloys in a partial or complete separation of the melt
 - Four promising candidate combinations have been identified where no dewetting was observed when bringing the material above the melting point



Dewetting of the material

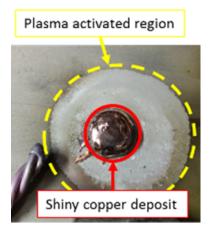


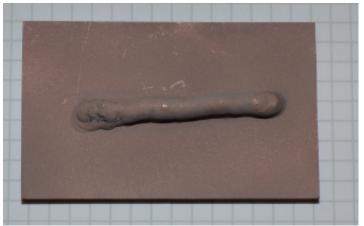
Sample without dewetting





- Approach Minimize the oxidation of the matrix by using in protective/reducing environment
- Severe oxidation of the matrix was observed using of argon as shielding gas
- High flow of argon gas solves this issue but causes a loss of the diamonds
- Forming gas gives very good results but more difficult to handle and hydrogen can result in a pore formation in copper





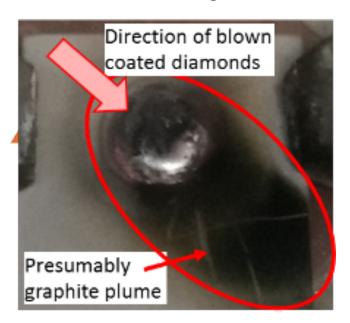






Challenge 6 :reduction of the thermal impact/apply sufficient high shielding gas to protect the diamonds

- Approach: reduction of the thermal impact is required on the diamonds
- Feeded diamonds are graphitized => coating can help to protect them
- Reduction of temperature required or using higher gas flow
- High shielding gas to protect the diamonds=> in this case severe overspray is observed.
- Additionally turbulences by the gas flow create irregularities in the distribution
- Dedicated argon box would be required to solve this issue

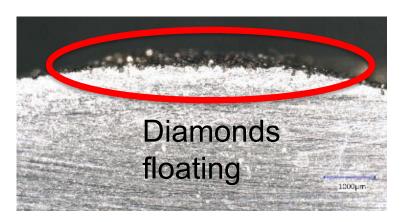


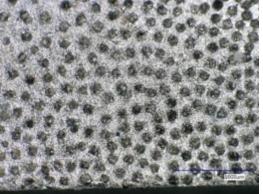




Challenge 7: Homogeneous distribution of diamond particles

- Approach: A good wetting is important to obtain a continuous arrangement of the diamond particles in the metal
- Diamond floating is a problem if a good wetting cannot be ensured
- Trials were made to minimize the difference between the density of the matrix and the diamond when using Aluminium and diamond with similar density;
- Floating of diamonds to the top was observed.

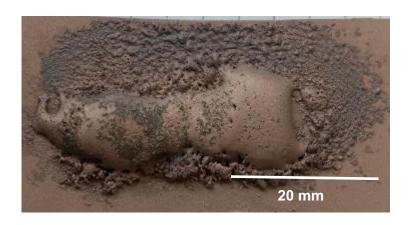






Challenge 7: Homogeneous distribution of diamond particles

- In copper diamond a similar behavior is observed
- Since the same effect was observed in Al-diamond it cannot be linked to the specific density or ratio of densities between the metal and the diamond
- Wetting is here also one of the main issues => results from # 4 have been applied using the combined approach of coating + copper alloy to have a sufficient wetting







Challenge 8: High diamond loading (>30 vol%)

- Approach: The control of the loading and finally the homogeneity is a difficult issue. The diamonds are floating to the top layer where a high concentration is observed.
- Wetting is important and be improved by changing the alloys.
- Using high content of diamond volume concentration (>50 vol%) results in a dilution down to 30 vol% if additional matrix material is used
- Bonding to the substrate is an issue; needs a good control of processing parameters to avoid a deformation of the substrate

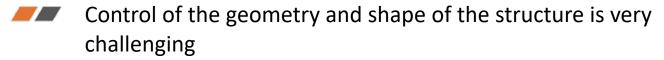








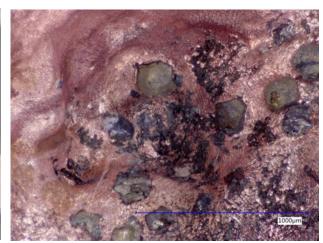




- High temperature/power is required to enable good wetting of the diamonds but this results in a loss of the shape
- Eventually controlled cooling/heating of the substrate is required to have more stable temperature conditions.







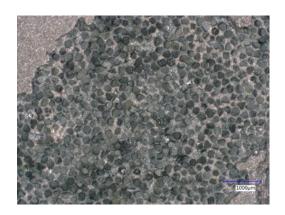


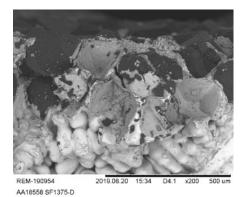


Experiments from wetting tests

- Based from experiments from wetting tests using a shape/mould it is possible to keep the shape
- Surface finish is still an issue and needs to be controlled in a better way







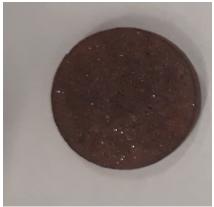
TECHNOLOGY



Preparation of samples using moulds to control the shape

- Using combined materials (coated diamonds + alloys) and performing tests in supportive moulds a good shaping was possible
- Different heating sources have been used
- Densities of the samples around 85-95%
- Certain loss of copper, therefore concentration might be higher than 50 vol%





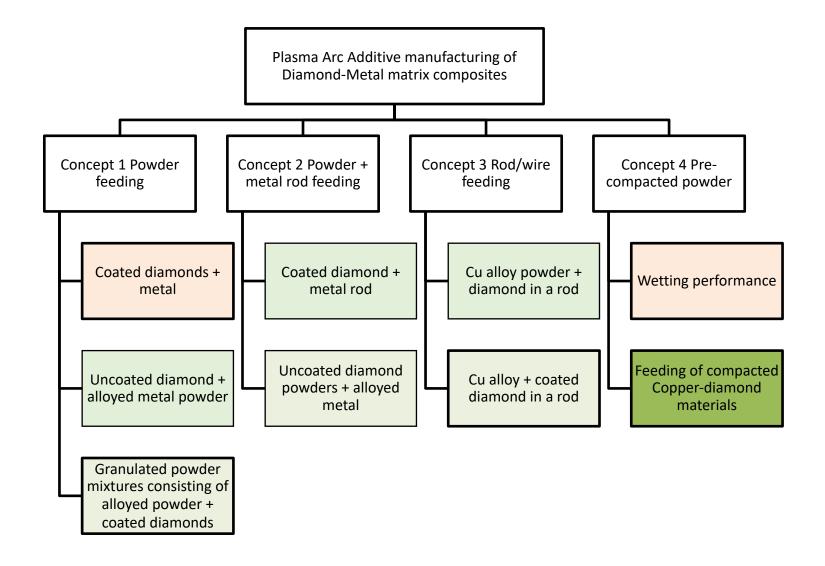




ID	Dicke	kappa	T100	Specifi heat	Archim. Density	Thermal Conductivity
	[mm]	[x 1e-6 m ² /s]	[K]	J/kgK	g/cm3	W/mK
15	3,98	182,34	0,35	0,40	5,26	384,6
16	3,64	152,44	0,45	0,40	4,46	272,6
17	4,50	261,67	0,34	0,40	4,65	487,9









Conclusions (I)

- Several challenges to solve when using a plasma arc deposition process for the manufacturing of a metal diamond composites
- Concept was developed to separate the different issues and to tackle them individually
- Main difficulties: many parameters which are severely linked to each other and which result in a contradiction, e.g.
 - High gas flow required for protection of diamonds => severe overspray observed
 - High temperature of the melt and promote wetting => degradation of the diamonds
 - Reduction of temperature/time with melt => less wetting/week interface and pores
 - Using of hydrogen to reduce metal matrix => creation of gas bubbles in the matrix
 - High energy required to enable a good bonding to the substrate low enough to limit the deformation/loss of shape of the substrate
 - ...and many more



Conbclusions (II)

- A granulation process for highly loaded diamond contents could be established
- First small sized demonstrator parts (20-30 mm) could be fabricated. An upscaling and optimization of the process is required (improvement of density)
- A "free-form processing" with this concept was not possible, so tooling is required.

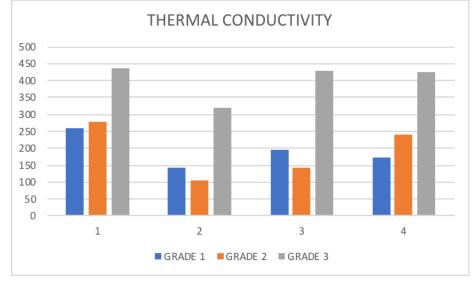


CTE < 10 ppm/K was achieved.





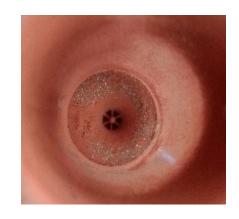






Know-How generated including spin-off

- Development of a concept for a powder feeder allowing to feed diamond containing material in different size => meanwhile we have identified a commercial powder feeding system which is on the market available
- For the Plasma based process new alloys have been identified which have an excellent wetting => this knowledge is used for the powder metallurgical processing
- Additionally based on the experience a liquid phase processing concept was derived and has been applied on prototype level for electronic components => currently under evaluation

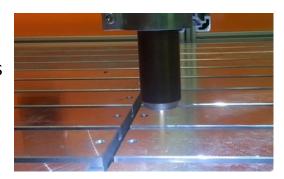




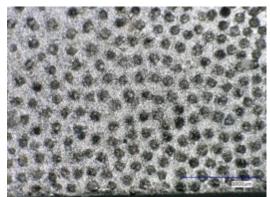


Potential commercialisation developments in the future

- Upscaling of the processing technology and demonstration
 - Main focus is to enable a fast processing for the heat sink manufacturing
 - Preparation of a plasma torch setup which allows to process pre-compacted of larger size
 - Oxidation has to be avoided, which means a setup which allows to deal with forming gas is required (encapsulated) and where the oxygen level is monitored



- Transfer of the developed compositions to the powder metallurgical manufacturing process for the heat sink preparation => application in thermal management applications (especially large structures)
 - The developed building blocks (combination of coated diamonds with alloys) allows a much faster processing of the materials
- Alternative applications might include the use the manufacturing in grinding tools => especially the arrangement







Suggestions for changes/modification of the funding scheme

- In general the PoC represents a good way to assess an innovative concept with a certain specific risk
- It allowed to assess different manufacturing concepts to identify the most promising concepts for follow up activities
- Overall administration/reporting is in a good balance, therefore we can recommend this funding scheme.
- Suggestions for improvement are:
 - Enable the possibility to proceed with follow-up activity (e.g. demonstration activity/"phase 2") after the proof of concept in order to increase the TRL, upscale etc.







Thank you for your attention!

MATERIALS. PERFECTLY. TUNED. Erich Neubauer e.ne@rhp.at

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