Material Issues of the Metal Printing Process, MPP

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Abstract

The metal printing process, MPP; is a novel Rapid Manufacturing process under development at SINTEF and NTNU in Trondheim, Norway. The process, which aims at the manufacturing of end-use products for demanding applications in metallic and CerMet materials, consists of two separate parts; The layer fabrication, based on electrostatic attraction of powder materials, and the consolidation, consisting of the compression and sintering of each layer in a heated die. This approach leads to a number of issues regarding the interaction between the process solutions and the materials. This paper addresses some of the most critical material issues at the current development stage of MPP, and the present solutions to these.

Introduction

The area of automatic Additive Manufacturing in metals comprehends an array of different processes and technologies. In the present situation, the practical applications of this "family of technologies" include such demanding items as small parts tooling, surgical implants and other highly specialised end-use products [1]. However, there are applications, where the contemporary commercialized processes are insufficient and the conventional additive process approaches limits the potential for competitiveness and further development [2]. This is particularly clear for products where size and building rate are critical, as well as the accuracy and the resolution, or when the application involves parts made of complex, or otherwise demanding materials. For these types of applications, a new process approach is required to further enhance and exploit all the potential advantages of Additive Manufacturing.

In response to this need, the Metal Printing Process, MPP, a novel Additive Manufacturing process is under development at SINTEF and NTNU in Trondheim, Norway [3], [4]. In difference to conventional Additive Manufacturing processes MPP uses a combination of an advanced layer fabrication system, with a subsequent metallic consolidation step. The layer fabrication is based on electrostatic attraction of powder materials, primarily metals and CerMets, to a charged surface [5]. The metallic consolidation applies the combination of pressure and heat to form loose particles into solid objects [6]. Each layer consists of two basic types of material; part material and support material. The part material is fused layer by layer into the final product during the consolidation process step, while the support material provides the necessary support, but otherwise remains unaffected during the process. After completion of each build, the support is removed to be recycled in future build packages. As with all Additive Manufacturing processes MPP comprehends a number of critical issues regarding technical solutions in relation to material interaction. The process cycle, from the layer fabrication to the heated compression during consolidation of the part, proposes challenges in terms of, design and material selection for the components as well as the behaviour and properties of build- and support materials. This paper describes the basic principle of MPP while it addresses some of the most critical material issues at the current stage of development and the present solution to these.

The Metal Printing Process

The principle of the process

The basic philosophy behind MPP is that fabrication of each layer in a separate system, prior to the layer-wise consolidation, makes it possible to engineer advanced multi-material layers, in regards to material position as well composition. This layer composition is fixated by the accelerated densification process, where each layer is exposed to a mechanical pressure-pulse during the deposition and sintering in a heated die. The time consuming single point sintering, or melting, that is predominant in commercial Additive Manufacturing systems for metals is thereby avoided [7].

The MPP principle consists of two main parts separated into two systems; the layer fabrication system (LFS) and the consolidation system (CS). (*See **Figure 1***)

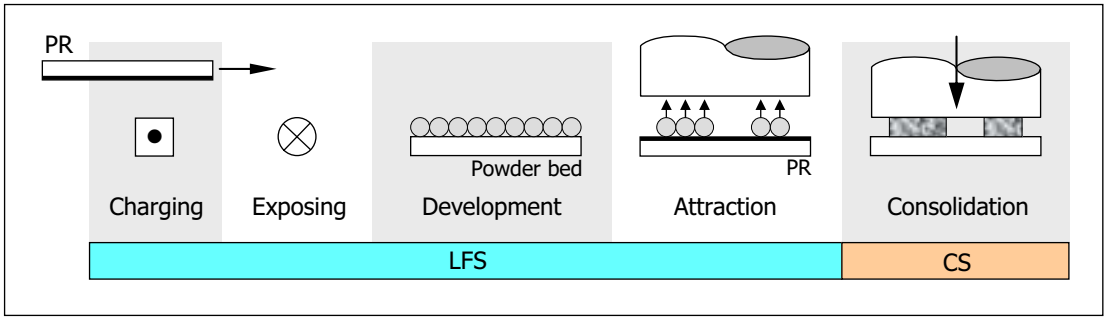

***Figure 1**: Principle schematic of the MPP sub-processes, linear machine concept*

Layer Fabrication

In the LFS, electrostatic fields play a fundamental role. The interaction of light with an electrically charged photoreceptor (PR) creates an electrostatic field pattern that varies in a one-to-one manner with the intensity of the exposing light. The technique for attracting powder particles onto the PR relies on the Coulomb's force that works on charged bodies in electric fields. When the charged PR passes over the powder bed, an electrostatic field will form between the charged areas on the photoreceptor and the powder bed. The electrostatic field exerts an attractive force towards the PR on the powder particles. When this force is strong enough to lift the powder particles, they will attach to the appropriate charged position at the photoreceptor's surface. Multi-material layers can be fabricated by adding more exposing units and powder beds to the LFS.

For metallic, and other conductive part materials, is applying an electrical charge to the powder particles fairly straight forward. However, since the support material should be inert to the part material(s) and otherwise unaffected by the heat during the consolidation, a ceramic nonconductive material composition is required for this purpose. To apply a sufficient electrical charge to nonconductive support material particles is a major challenge for the development of MPP, and at the present stage there is no final solution to this; however there are several possibilities under investigation. One solution to this, which finds it's inspiration in the copier technology, would be to use tribo-electrical charging by means of rotating the support powder with a coated ferromagnetic carrier powder. The charged carrier and support powder blend is then transported within distance to the charged PR to create the sufficient electrical field for the transfer of the support material particles into position in the powder layer, while the carriers are held back by magnets. (*See **Figure 2.***)

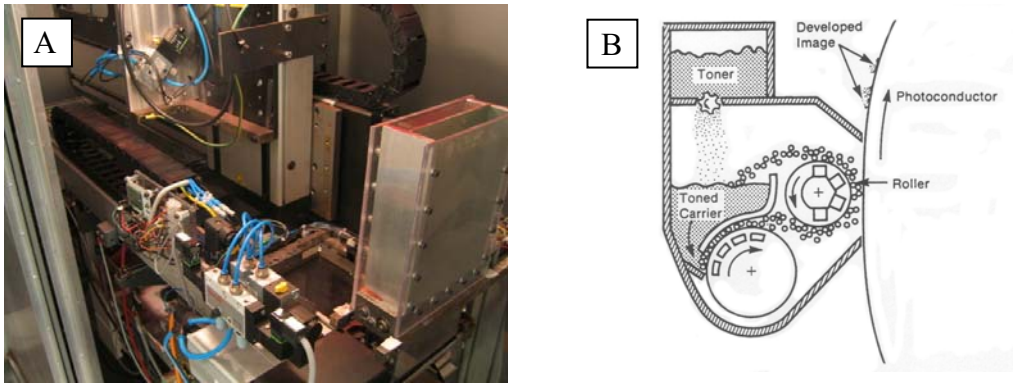

***Figure 2**: (A), the layer fabrication system from the MPP demonstrator machine. And (B), the suggested system for developing a layer form irregular nonconductive powder particles, where "toner" corresponds to the support powder. Picture from [8].*

Consolidation

After fabrication, the complete layer, including both part materials and support, are transferred from the PR to the punch by electrostatic forces. Then it is moved with the punch to the consolidation system, CS where loose powder layers are converged into useful engineering components by compaction and sintering in a heated die. The compaction relies on an external source of compressive power for deforming the powder into a high density component that (layer-wise) approaches the final geometry. In the MPP system only thin layers, -each with a thickness of a single particle size, are compacted at a time [7]. This simple, two-dimensional shape means that single action pressing (the pressure is transmitted from one direction only) is enough to ensure good packing properties while maintaining dimensional control.

The sintering, and thus the formation of metallic bonds in the parts, relies on the joint effects of the compression and elevated temperature in the die. The active deformation of each particle in the added layer does not only increase the contact surface between the added particles and the previous layer, it also increases the dislocation density in the material and thereby provides several new nucleation points for recrystallisation, and thus enhances the sintering activity in the material. Therefore the sintering temperature can be kept considerably lower than under more "normal" circumstances, (-that is, without pressure), and still reach full, or desired density in the part.

The consolidation unit consists of the previously mentioned punch and a die surrounded by an induction coil. The die is heated and held at a constant temperature during the entire build time. To prevent oxidation, a protecting atmosphere is applied during sintering. The combination of heat and pressure during compaction and sintering puts special requirements on the materials and design for these parts. First of all, the punch and die must withstand the temperature and pressure without deformation during consolidation of each new layer. Furthermore must the fitting of the punch within the die cavity be precise in order for the compressed material not to squeeze into the gap between punch and die wall and thus jam the punch's movement, meanwhile thermal expansion of a tightly fitted punch would have the same effect. In order withstand the pressure while maintaining minimal thermal expansion makes cemented carbide the natural choice, and to secure that punch and die are within a secure temperature interval are the external of the die as well as the punch water-cooled.

However, the cobalt matrix in the cemented carbide material is known to be reactive, and would adhere to several technically interesting part materials, such as iron (steel), copper, and

titanium, at the desired process temperature interval. In order to avoid this are the surfaces of the punch and die coated with a thin layer of alumina [7]. (*See* ***Figure 3.***)

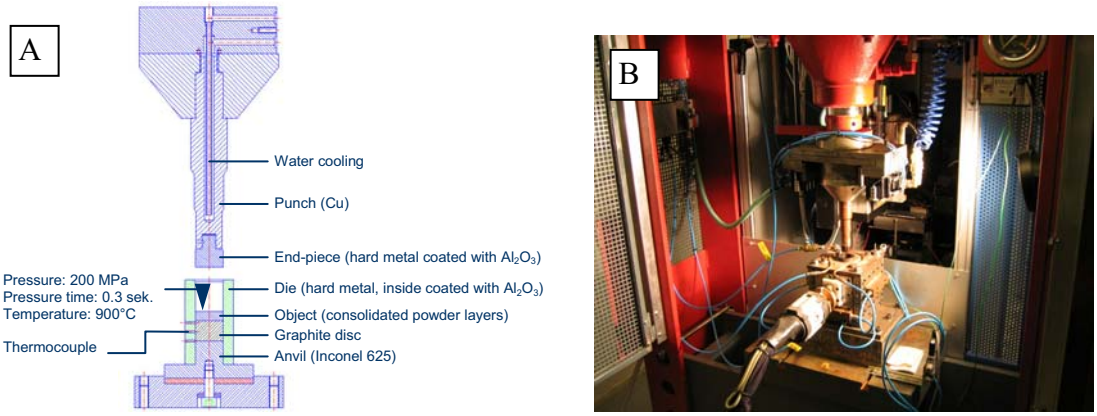

Figure 3: *(A), the consolidation unit in principle, with exception for the heating and cooling units for the die. (B), the same unit in reality, (including the heating and cooling units) in the present set-up of the MPP demonstrator.*

Part Materials

Layer-wise addition by compression

As stated earlier, the Metal Printing Process uses two principal types of powders: a part material powder which consists of the powder(s) that forms the material which the component is made of, and a support powder, that provides the necessary support to build shaped components. After the layer has been fabricated in the LFS and transferred to the punch, the part-, and support, powders are pressed by the punch onto the building surface in the die. In this part of the process, the part (and support-) powders are transferred from the punch to the component as the part materials adhere to the component's surface. The yield of this transfer is dependent on a balance of the forces affecting the powder during the compaction. If the compaction is made below sintering temperature, and if the punch is covered with an electrically insulating ceramic layer, then the charged powder particles are attached to the punch by electrostatic forces alone. However, if the punch is electrically insulated with a wax, then the powder particles, in addition to the electrostatic forces, also are attached with the adhesive effect of the wax. (The wax also has the effect of a lubricant during the deformation of the particles, and inhibits cold welding of the particles to the punch. The remaining wax on the powder particles' surface will be burned off during sintering as binders do in conventional powder metallurgy.) [7]

Contrary to the forces binding the powder to the punch are the forces that are connecting the powder to the building surface. For the support material it is only a matter of green strength, but for part materials there is, during compression below sintering temperature, also an interlocking of the deforming particles. The magnitude of this effect is to a large extent dependent on the type of powder used. A powder which has been used extensively during the development of the Metal Printing Process is Höganäs ASC200 iron powder (200 mesh). This highly irregular shaped powder, (*See* ***Figure 4A.***) has the advantage for cold consolidation that the particles can easily interlock with each other when a new layer is pressed into the die. Moreover, since this iron powder contains only small or insignificant fractions of alloying elements, it is also easy to compress to a high density, (*See* ***Figure 4B.***) [7].

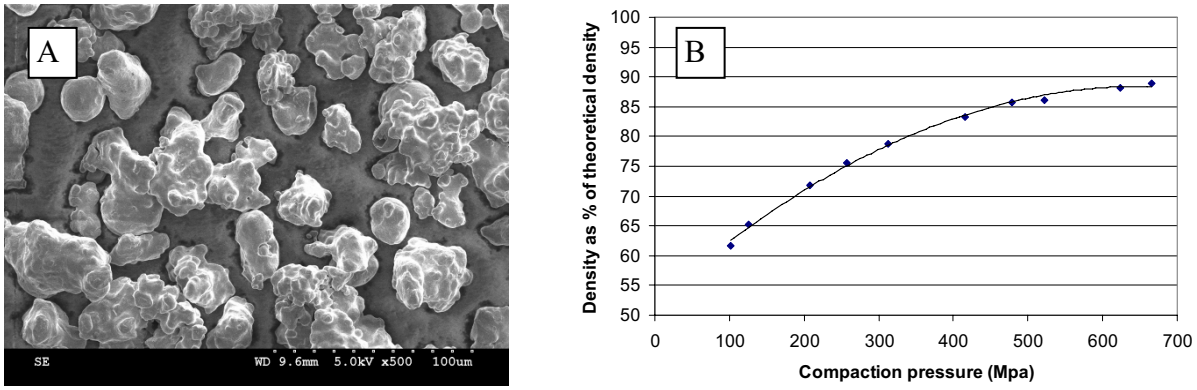

Figure 4: *(A) SEM secondary image of ASC200 iron powder used in cold compaction in the MPP demonstrator. (B) Cold compressibility of the same ASC200 powder.*

Other powder materials have also been used successfully for cold compaction in the MPP layer-wise additive process. Among them are copper and aluminium powder. These have been water- or gas atomized powders with a far more spherical shape than ASC200, however for these materials the success in adhering the new layers to the building surface may be attributed to other phenomena such as cold welding of virgin (non-oxidised) surfaces exposed by the extensive deformation of the particles.

In the situation of consolidation above the sintering temperature the conditions are quite different. During the compression of the new layer onto the heated building surface, the deformation triggers instant sintering of the new powder particles onto the previous layer. However, this approach requires that the part is being kept at an elevated temperature for the whole building session, and this may, dependent on which temperature the build (i.e. the part and support in the die) is kept at, and for how long, cause grain growth and thus have a negative effect on the material properties of the final product. Running the process continuously at an elevated temperature also puts greater demands on the material properties of the equipment. However, since it is most desirable that the process produces fully functional parts without the need for post-processing, the continued development of MPP is primarily aimed at consolidation above sintering temperature, despite the challenging issues [7]. Meanwhile, for research and development purposes, layer-wise cold compression is a way of getting insight in the materials' behaviour during the process.

The layer-wise consolidation in the MPP seems also to affect the material properties of the material. Sintering of layer-wise cold compacted samples have shown significant strength increase as compared to conventionally processed powder materials (*See* ***Figure 5.***). A plausible explanation for this improvement is that the compaction in thin layers eliminates much of the internal friction in the powder which resists the densification, and that the extensive deformation of the particles during compression increases the dislocation density and provides more nucleation points for new crystals, thus producing a smaller particle crystalline structure in the material. This will be the subject for continued study throughout the development of MPP.

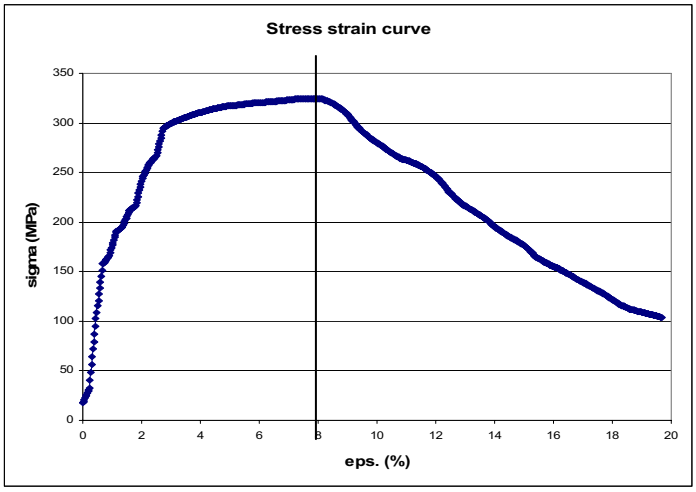

Figure 5: *Stress-strain curve of cold compacted and sintered NC 100.24 iron powder from Höganäs. The yield stress is 320 MPa, and the eps. 11-13%. Better than stated in Höganäs mechanical properties booklet for this material.*

Influence of powder particle size

Since the layers that can be fabricated on the PR (photo receptor) only can have the thickness of one particle, the building speed of the MPP has a linear dependence on the particle size of the used part material powder (*See* ***Figure 6A.***). However, the accuracy and resolution of an additive process cannot be more precise than the size of the units added, which for MPP is the particle size in the part material powder. Therefore the accuracy will have a similar linear dependence as illustrated in ***Figure 6B***.

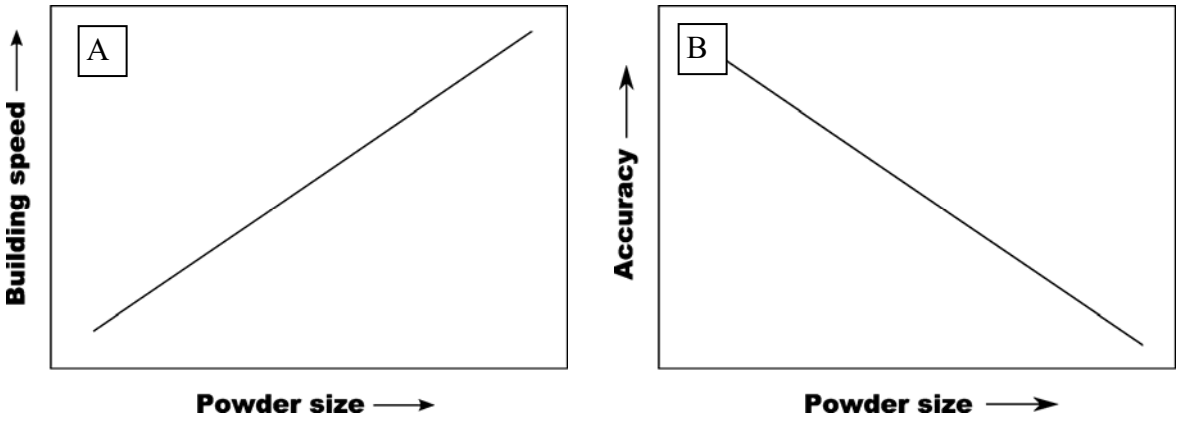

Figure 6: *Illustration of the influence of powder size on A) the building speed and B) the accuracy of the final object*

However, since each added powder layer is compressed in the z-direction, the accuracy and resolution in the x-y-directions will be much more affected by the particle size than in the z-direction. Moreover, since MPP, in difference to most commercial Additive Manufacturing processes, adds, -and consolidates, the material a whole layer at a time, it has the potential to be by comparison a very fast process. Therefore there is a more than reasonable possibility to optimize these parameters while maintaining the process capacity in regards to building speed and accuracy on a competitive level.

Multi material components

One of the advantages of the combination of a separate layer fabrication system and the pressure-sintering consolidation which is the fundamental principle of MPP, lays in the greatly expanded possibilities to produce components in two or more materials. Since the layers are fabricated separately, and there is no melting of the material during the consolidation, there are more alternative methods to engineer the material properties in the part available. For example:

1. Two, -or more, different part material powders: A component could, for example be built with one part made of copper and another of steel. This can easily be done by adding more exposing units and powder beds, in this example one with Cu and one with Fe, to the LFS (*See* ***Figure7.***).

Figure 7: *Multi material component made of iron and copper built with the MPP demonstrator.*

2. Two alloys in one component: Different amounts of alloying elements, for example C and Mn, can be added to pure base material part powder, for example Fe. After sintering and alloying element diffusion, this can give local compositional variations in the part, which lead to variations in the microstructure and thereby the mechanical properties within the same component.
3. Partial composite: The hardness and abrasion resistance of a component can significantly be enhanced by the addition of a secondary wear resistant phase. For example, an aluminium component with an addition of Al_2O_3 to the powder. This addition can be oriented only to the parts of the component where the improved hardness is needed since the addition of Al_2O_3 to aluminium usually comes at the expense of the ductility.
4. Reactive sintering in a part of the component: Addition of a reactive element to the powder can be used to produce objects with partial inter-metallic properties. For example the addition of a small fraction Al to Ni powder would result in the formation of Ni_3Al during sintering.

Clearly, there are many ways to exploit the possibilities of MPP, and there is little doubt that as long as MPP is a viable technology, there will be much more research to do in this area.

Support Materials

General

The primary advantage of Additive Manufacturing technologies lies in their ability to build complex parts in a single process step. The separate, advanced, layer fabrication system enables MPP to build parts in complex, variable material compositions. However, due to the layer-wise pressing and sintering process approach, the capacity for MPP to build parts with complex geometries is entirely dependent on designing a capable support material system. Such a system consists of the support material and the technical solution how to apply this material on the PR (photoreceptor) in the LFS (Layer Fabrication System).

There are a number of requirements the support material must fulfil;

1. The support must have a compression characteristic that is compatible with the part material. If the support material builds less (adds less dense powder to the z-direction) than the part material, it will not provide the necessary support during the compression of the following layers which would cause damage in the part. If the support material builds more (adds more dense powder in the z-direction) than the part material, then the compression of the layers will be insufficient and cause unwanted porosity in the part. (*See* ***Figure 8.***)

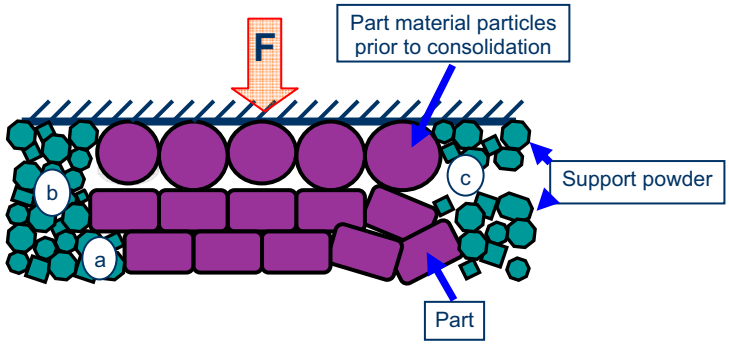

Figure 8: *Principal support requirements; a) must provide sufficient support under overhangs, where b) too much build, means unsatisfactory densification, and c) too little build means unsatisfactory support*

2. The support material must be chemically inert, be thermally stable, and have minimal adhesion to the part material, throughout the process temperature interval. In respect to this point, it is likely that full exploitation of the MPP technology for a wide array of materials will require more than one support material composition, but for the present development situation, a process temperature interval of 8-900 ºC, -up to a maximum of 1200 ºC, will suffice.
3. For the manufacturing of complex parts, the support must be easy to remove after the build has been completed.
4. Since it obviously is undesirable that the support material sinters during the consolidation process, but still must remain in the die, (and not returns with the punch after the compression,) it is also necessary that the compressed support have sufficient green strength. Furthermore, since it also is unlikely that so fundamentally different materials as the part material and the support material should have identical behaviour during compression, a high green strength in the compressed support material would also provide some additional robustness of the support during compression.
5. Finally, it is most desirable that the support powder can be recycled after the part has been completed, while it does not raise much dust in the air during processing and handling.

Apart from the principal functional requirements mentioned in point 1, the most pressing functional requirements for the selection of support material are the ones concerning the material's thermal and chemical properties, mentioned in point 2. In accordance to the requirements of chemical inertness and thermal stability, ceramic powders would be the obvious choice, and in respect to minimize the adhesion to most part materials in the present development situation, alumina has been selected as the primary component in the support material [9], [10]. However, ceramic materials have a fundamentally different electrical properties and compression behaviour compared to the, primarily metallic, part materials. Therefore will the most pressing issues in the continued development of the support material system be to produce a compression behaviour that is coherent with the part materials' and develop a delivery system for the support powder to the PR in the LFS. The remaining part of this section in this paper will focus on the investigation on the issues of the support materials compressive behaviour.

The behaviour under compression and green strength of alumina in various compositions has been investigated in a press set-up as seen in ***Figure 9***.

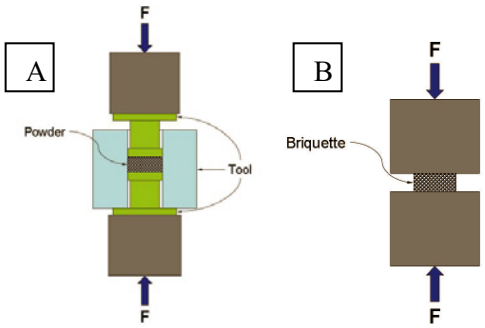

Figure 9: *The uniaxial press setup used to investigate powder compression properties and compressive green strength of the support powder. A) Powder is compressed to a briquette in a tool, 25 mm diameter, inserted in the press, while force and displacement is monitored. B) The compressive green strength is determined by measuring the force required to crush the briquettes.*

The maximum compressive force in the press is set to 25 kN in accordance to the present work pressure in the MPP demonstrator machine. Initial tests at this work load showed that it is not very likely to find compression behaviour, with any likeness to that of metal or any significant green strength in a pure alumina powder blend. Since the part materials are predominant metallic, they will show plastic deformation behaviour during consolidation. The alumina, however, will not deform under MPP process conditions, and the displacement during compression will consist entirely of particle orientation. Thus a fine particulate material that enhances particle movement and green strength while conforming to the other requirements mentioned, would be the ideal addition. Boron Nitride, BN, is a stable chemically inert ceramic material, which in its hexagonal molecular structure often is used as a high temperature lubricant [11], [12] (*See also* ***Figure 10.***). Since BN also has low adhesion to most relevant part materials, it is chosen as the secondary material for the support powder composition [7].

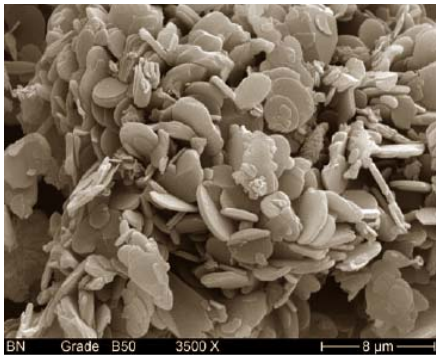

Figure 10: *SEM micrograph of hexagonal Boron Nitride, BN B50, from H.C. Starck Gmbh. The small particulate, disc-like crystalline structure typical of hexagonal BN, both ensures a low tap density, 0.3 - 0.5 g/cm³and a high specific surface area 4.0 – 6.5 m²/g (AREA METER II per BET). The alternative BN quality: BN C, are even smaller in particle size, and has tap density 0.25 - 0.5 g/cm³and a high specific surface area 10.0 – 20.0 m²/g (AREA METER II per BET).*

For alumina base material was AMPERIT® 740.8 with a particle size range as shown in ***Table 1.,*** from H.C. Starck Gmbh, chosen. Both BN and base material alumina were tested in the experimental set-up, (as seen in ***Figure 9.***) unblended as well as in various compositions. However, for comparison regarding the effect of particle size, was an alternative alumina powder (from ALCOA) with a smaller particle size (D_{90} = 11.04 µm) also investigated.

-44 µm	100 %
-31 µm	95 %
-5.5 µm	3.5 %

Table 1: *Grain size of AMPERIT® 740.8 (measured by Microtrac SRA 150)*

The behaviour under compression (displacement in relation to compressive force) and the spring back at the release of pressure for the different compositions can be seen in ***Figure 11.***

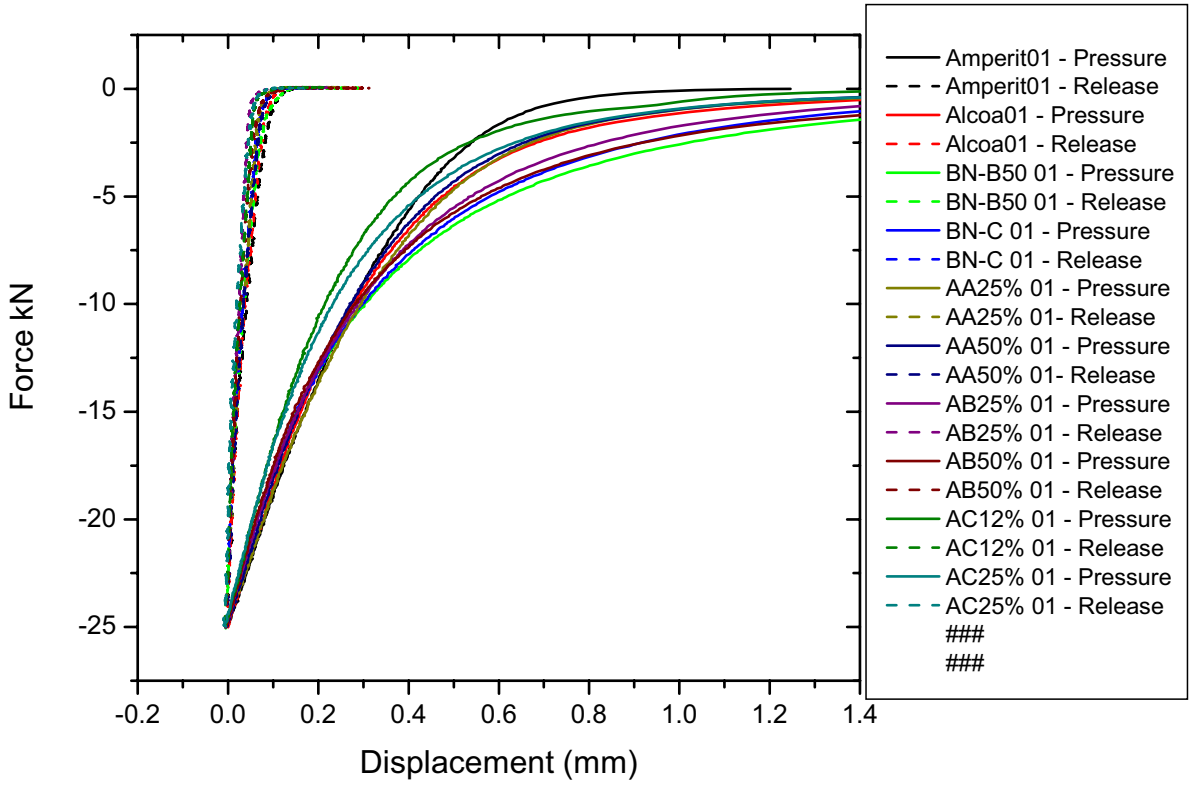

Figure 11: *Pressure and displacement of various support powder compositions. Unblended powders are represented by their full names while the different blends are represented by abbreviations, where "A" is the AMPERIT® 740.8 base material and the secondary component is represented by the fraction in weight%; AA25%= 25 % Alcoa, AA50%= 50% Alcoa, AB25% = 25% BN-B50, AB50% = 50% BN-B50, AC12% = 12% BN-C, AC25% = 25% BN-C. Zero displacement is set to the point of maximum compression.*

From ***Figure 11***, it is evident that the different support compositions all have similar compression behaviour as could be expected from dry compression of ceramic powders. However, some patterns are clearly deducible from the curves; unblended alumina powders have initially little displacement and a steep increase in force for little displacement towards the point of maximal compression. In difference to this, a plastically deforming part material would have much more sloping curve during compression and sintering. However a decrease in particle size leads to a more slanted curve with bigger displacement under compression as can be seen in the Alcoa curve. The most sloping curves are those with pure BN powder, in particular BN-B50, however the curve for AB25% is not far from them. Surprisingly, however not critical for this work, is that the curve for AC25% appears more like those for AA25% than AB25%.

From the compression curves, it seems quite clear that it can not be expected that the compression behaviour of part and support material will follow closely to each other, and therefore the green strength, and the extra robustness it brings to the support is of significant importance. The briquettes formed in the compression experiment were crushed in a set-up as shown in ***Figure 9.*** to evaluate the green strength (*See* ***Figure 12.***). Some powders (pure AMPERIT® 740 and some Alcoa), did not have enough green strength to be handled and were therefore omitted from the experiment.

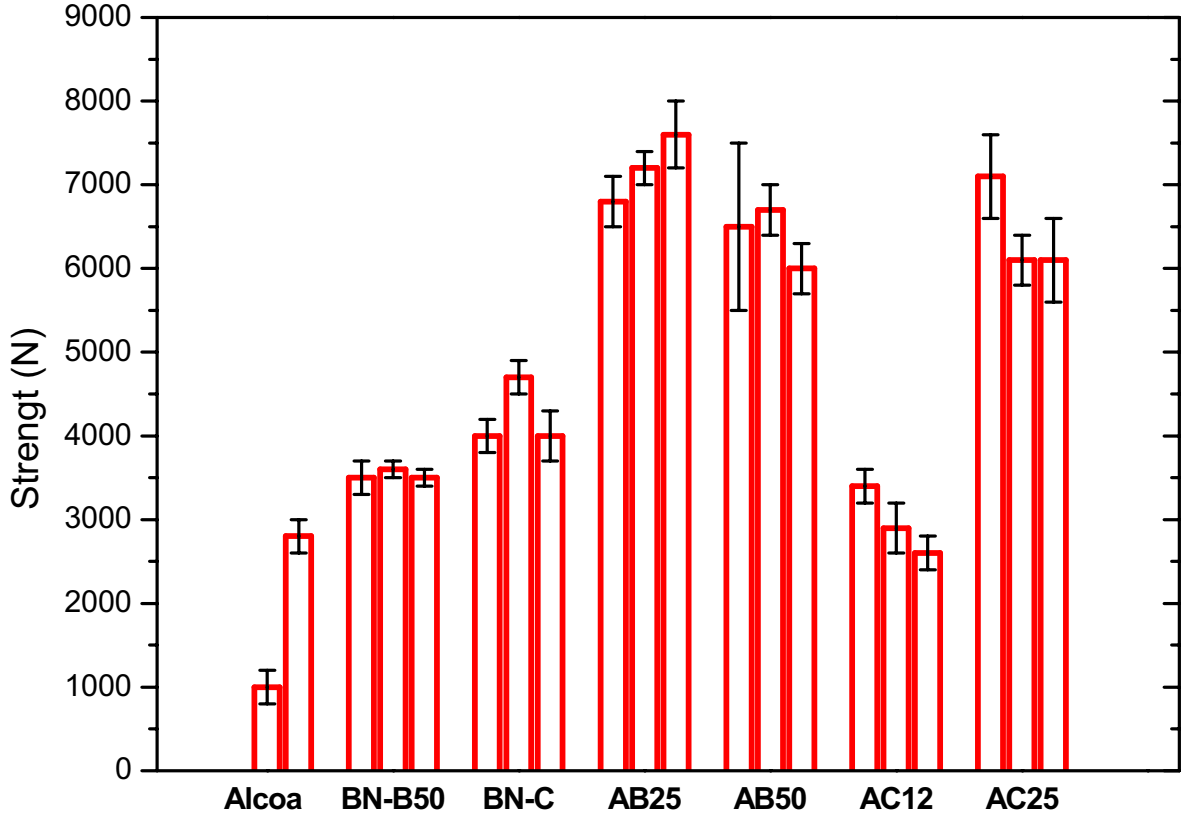

Figure 12: *Compressive green strength of various support powder blends as derived from the crushing of briquettes between parallel surfaces.*

The crushing behaviour of the support powder briquettes showed some surprising elements. Unblended powders, in particular Alcoa, had, as could be expected, a brittle behaviour, and yielded to the pressure at one instance, whereas the combinations of Alumna and BN, especially those with 25% BN, were almost plastic in behaviour. This could be explained by the formation and closure of micro cracks in the briquette during crushing. Supposedly, the lubricating effect of BN has aided a close arrangement of the alumina particles in the briquettes. As cracks are formed during crushing, and the planes of the cracks slide against each other, the particles in the cracks' surface are lubricated enough for rearranging themselves and form new Van der Waals' bonds with smaller loose particles moving between the surfaces. The cracks may close while new cracks are formed. The unblended powder briquettes on the other hand, are mostly held together by the elastic forces between interlocking particles in the powder mass and when that lock is broken during crushing there is little or no chance for the particles to rearrange themselves in an interlocking manner again. This "plastic-like" crushing behaviour in combination with the apparent high green strength of, in particular, the AB25% composition is encouraging and will be used as a basis for the continued development of the support material system.

Conclusive Remarks

The development of MPP is presently in a very dynamic phase. While the layer fabrication system has been concept proven, new and improved methods are being investigated for the fabrication of both part and support materials. The very promising results that has been reached with the layer-wise compression and sintering, leads to further investigation of this type of consolidation for a wider selection of materials, and material combinations. The development and integration of the support material system is making significant progress with material compositions and machine design. However MPP is by comparison a complex approach to Additive Manufacturing and many issues remain unsolved or are subjects for optimisation. There is great potential for interesting and challenging R & D in the years to come.

Acknowledgements

The research and development of MPP is being financed by grant from the Research Council of Norway and "Custom-Fit, a project under EU's 6th Framework Program.

The authors wishes to express their gratitude to the other members of the MPP research core team, in particular: Olav Åsebø, Martin Syvertsen and Terje Mugaas. "If you guys didn't do such a good job, we could never write such long papers."

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