Powder metallurgical steel quality by additive manufacturing using VAT polymerisation technology

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Abstract

VAT polymerisation of metal components is economically viable with a resulting high material quality. While the 3D shape is realised by photo initiated curing, penetration depth of light in the slurry is critical for a reliable and economic feasible process. This has been solved by optimising the slurry composition in combination with a printer capable of applying thin layers with micron accuracy. Debinding and sintering of the finished product is thereafter equal to existing MIM processing, resulting in stress free high density metals as shown for 316L stainless steel.

Contents

Preface

Injection moulding is a proven and reliable way of producing powder metallurgical or ceramic parts. A carrier polymer is used for the shaping of a powder compact. After shaping, the polymer is removed by a debinding step and the finished product is realised by sintering of the resulting body of metal or ceramic powder to a dense component. In 2013, ECN and Admatec developed a Digital Light Processing (DLP) or VAT polymerisation AM process for alumina, zirconia and silica ceramics. After this successful development of printing translucent powders via DLP, research was continued to use this technology for non-translucent or light absorbing powders like black ceramics and metals. After 3 years and therewith a 3 times longer development route, the technology is proven with 316L stainless steel and a prototype Admetalflex printer has been built and presented.

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Introduction

The Digital Light Processing (DLP) technique, a photo polymerisation based additive manufacturing technology, projects a bitmap image onto a layer of photosensitive resin via a light engine and thereby curing it. This process is repeated layer-by-layer to obtain a complex 3-dimensional product. Both metal and ceramic powders can be mixed with a photosensitive resin, resulting in a composition containing approximately 50 vol% powder. By using this slurry a composite part existing of metal/ceramic powders embedded in polymeric solid can be obtained. Later in the post processing steps, the polymer can then be decomposed and removed, leaving a metal/ceramic component which then is sintered, see **Figure 1**.

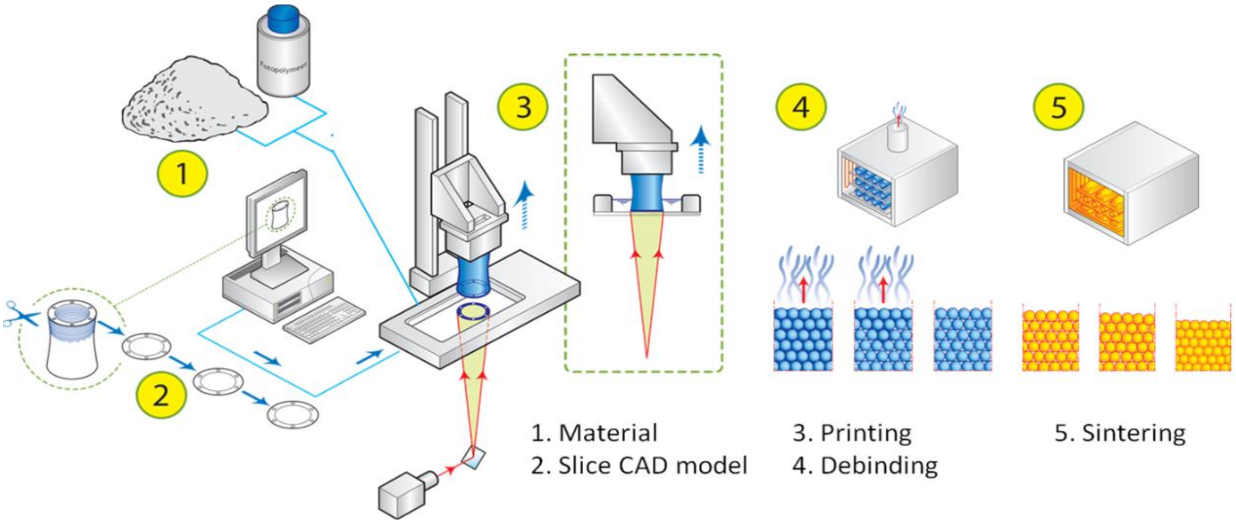


Figure 1: Process of DLP printing of ceramics or metals.

For DLP the thickness of the cured layers should be enough to get an economically viable process. In practice 20 µm or more is used. For non-translucent or even light absorbing powders, therefore, two main problems have to be solved:

1. How to increase penetration depth of the photons
2. How to realise photo Initiation and formation of radicals for polymerisation

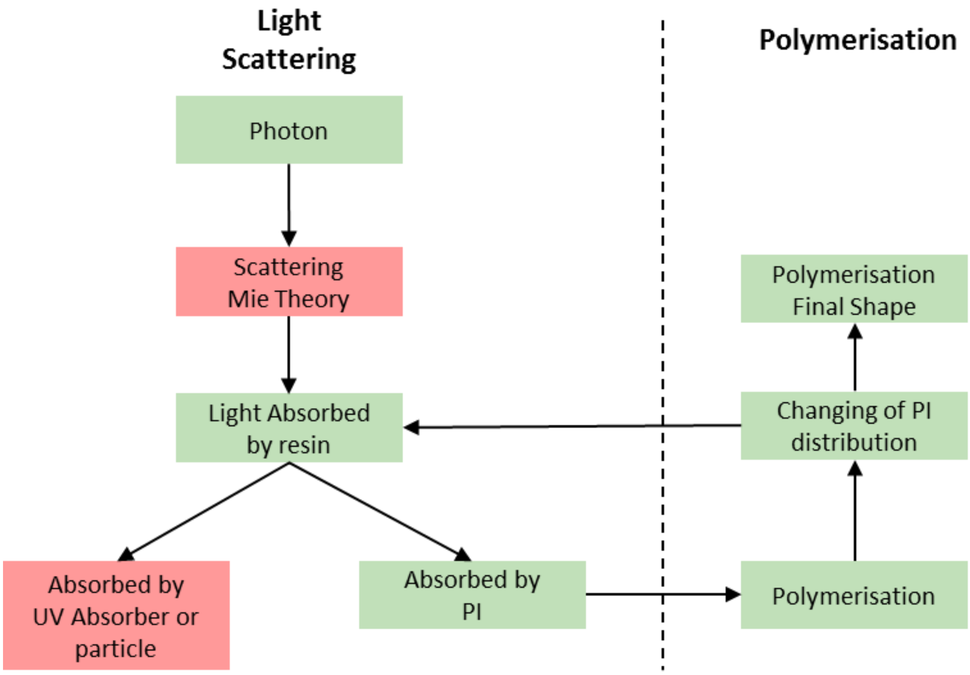
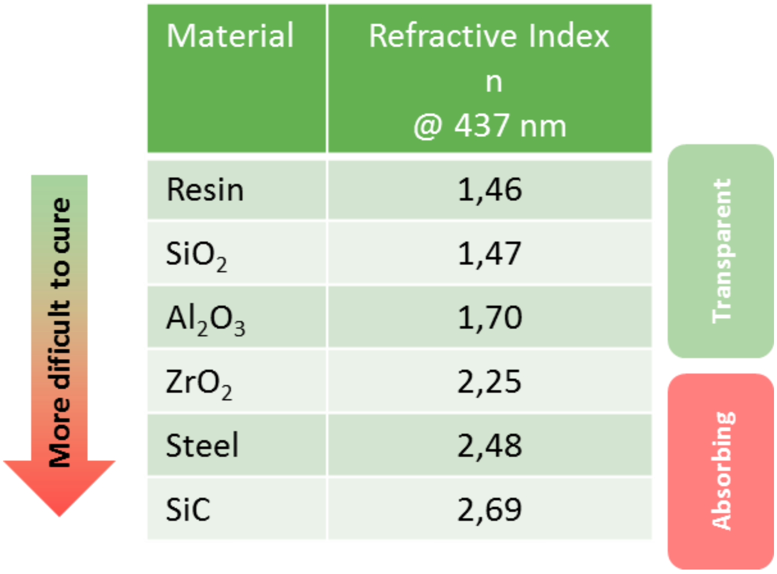


Figure 2: Different aspects of light penetration in slurries for the DLP process.

The penetration of light into a slurry is influenced by the light absorption of the used powder and by the scattering, due to the powder:

- The light scattering increases by the difference in refractive index between the resin and the used powder. Severe scattering leads to limited penetration depth and also over-exposure at the edges of the printed body, affecting dimensional control.
- Light absorption decreases the light intensity and the penetration depth of the minimal intensity needed for proper curing.



Material	Refractive Index n @ 437 nm
Resin	1,46
SiO_2	1,47
Al_2O_3	1,70
ZrO_2	2,25
Steel	2,48
SiC	2,69

Figure 3: Reflective index of powders and base resin.

The penetration depth of light in a slurry with a monodisperse particle size distribution increases with the particle size, **Figure 4**.

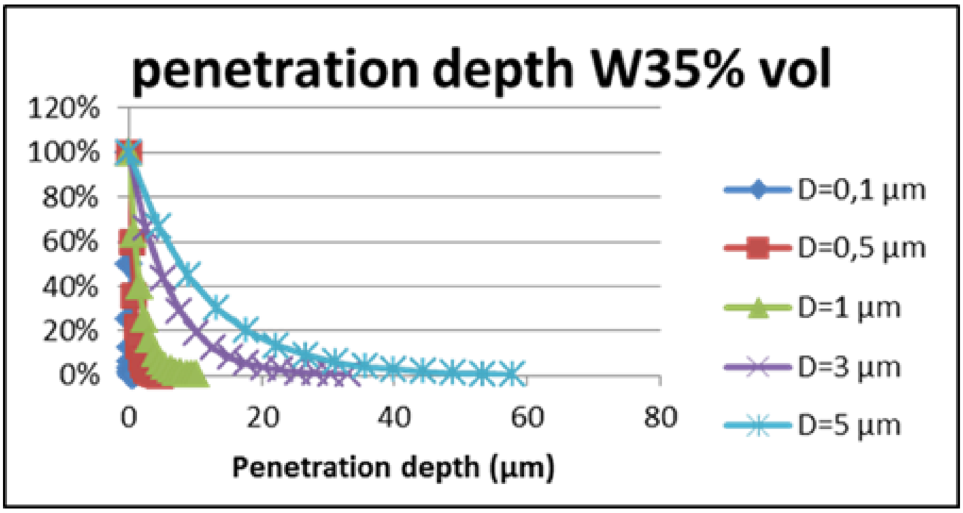


Figure 4: Penetration depth of light depending on particle size of absorbing powder.

The drawback of increasing particle size can be the reduction of the sinter ability of the composition. For ceramics, typical micron or submicron powder is needed to sinter too dense products. Most metals, however, will also sinter to a dense product with more coarse particle sizes. For stainless steel MIM typical 13 µm particle size powder is used to produce dense products.

Proper curing will be obtained by an optimum between the slurry composition and used dose of curing light. As the dose is the multiplication of light intensity and curing time, the same dose can be set with either a high intensity / short time or a lower intensity / longer time. When applying very high intensities however, the product accuracy will be reduced, due to the scattering of the light.

By optimising these settings, 316L stainless steel can be printed with 20µm layers in green state with doses of about 150 mJ/cm^2.

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Properties of printed metal

Afterwards, the approximately 50 vol% of polymer has to be removed during debinding. Special attention should be payed to the removal of the residual carbon to prevent carbon dissolving in the steel matrix, which will be detrimental for the physical properties. The chemical composition after sintering of the material is shown in **Figure 7**. The overall chemical composition is determined by EDX, while the carbon content is determined separately (by Tata steel). As can be seen, the chemical composition is well within the specifications and the carbon content is very low **Table 1**.

In **Figure 6** the elemental mapping of the major elements present in 316L are shown. The mappings show the surface morphology (from the valleys only limited information will be received, which explains the dark areas in the mapping for all elements) and also show that there is no segregation in the material. Also very positive is the fact that almost no oxygen is present, hence no chromium oxide is formed.

With proper settings of the sintering step, the sintering results in good shape retention (for instance straight corners, see **Figure 5**), a regular equiaxed microstructure with a grain size of 50-150 µm and a measured density of at least 98%.

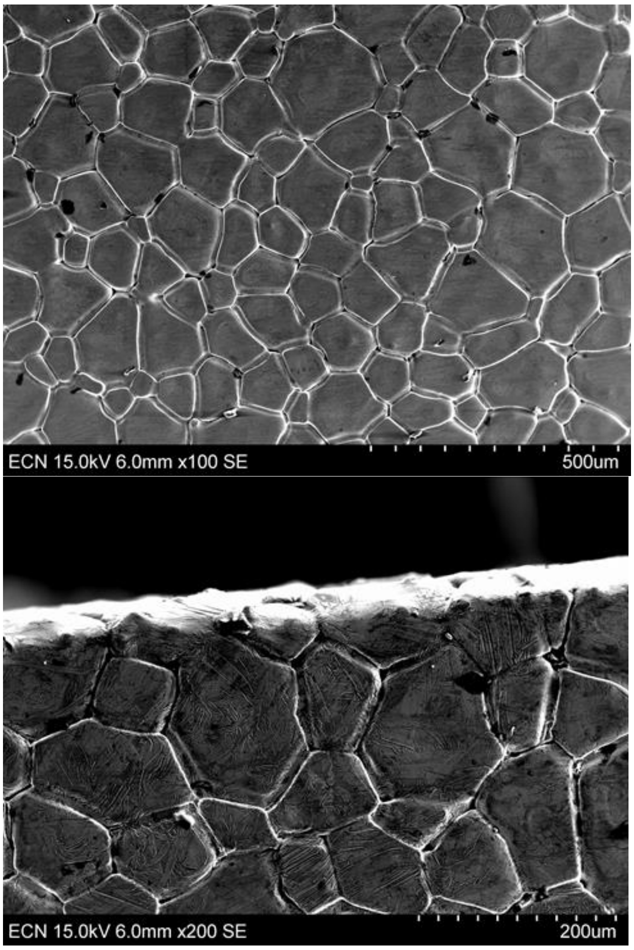


Figure 5: Microstructure of sintered DLP formed 316L stainless steel.

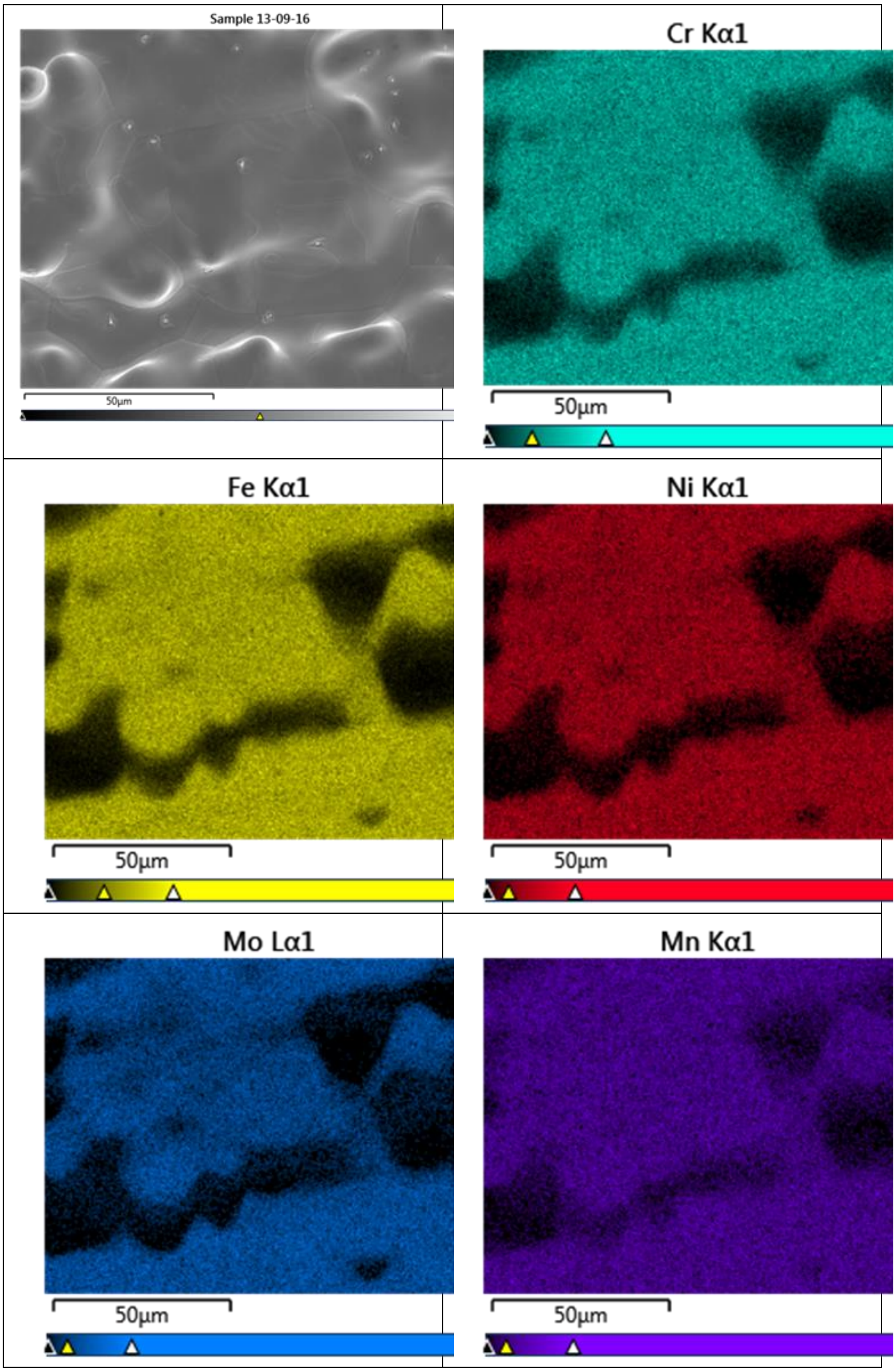
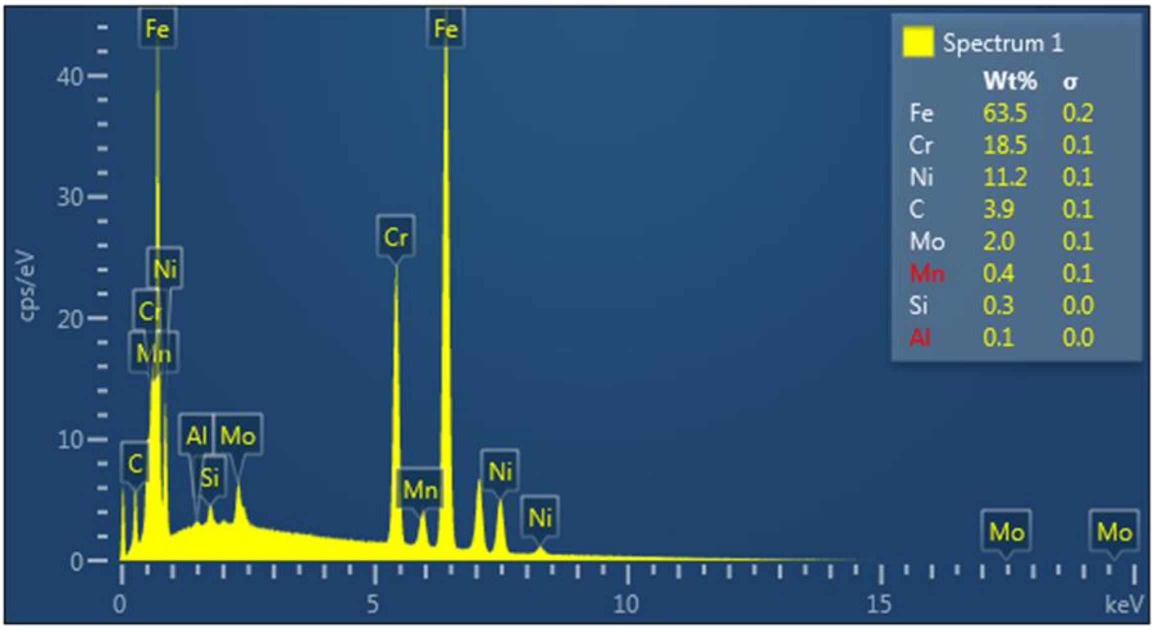


Figure 6: Element mapping of the sintered S316L end product.



Spectrum 1

	Wt%	σ
Fe	63.5	0.2
Cr	18.5	0.1
Ni	11.2	0.1
C	3.9	0.1
Mo	2.0	0.1
Mn	0.4	0.1
Si	0.3	0.0
Al	0.1	0.0

Figure 7: Composition of a sintered 316L DLP printed body.

Table 1: The overall chemical composition of sintered 316L compared to material specification.

	Specification [%]	Measurement (3 separate measurements) [%]
Carbon, C[1]	<- 0.030	0.002
Chromium, Cr	16 – 18	17-19
Iron, Fe	As balance	As balance
Manganese, Mn	<= 2.0	0.4
Molybdenum, Mo	2.0 – 3.0	2.1 – 2.2
Nickel, Ni	10 – 14	12 – 13
Silicon, Si	<= 1.0	0.2 – 0.4
Phosphorous, P	<= 0.045	Not determined
Sulfur, S	<= 0.030	Not determined

Note: C is determined separately at TATA steel.
Other elements are determined with EDX.

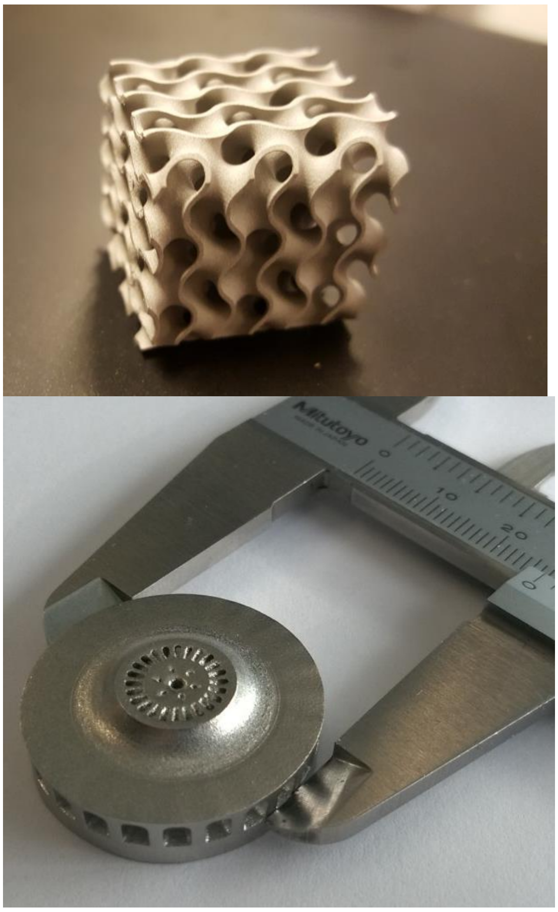


Figure 8: Printed metal products.

For printing of ceramics the Admaflex 130 is used. The drawback of the current machine design is, that it is optimised for printing of layers of 20 µm or more, applicable for typical optical penetration depths of 100 µm or more. For light absorbing slurry compositions with optical penetration depths of 40 µm or less, accurate thin layers are crucial.

The current solution is to press the workpiece in an excess of slurry until the intended layer thickness is achieved. The machine concept is therefore adapted to make thin layers applicable. In principal the idea of doctor blade casting is used to calibrate the resulting layer thickness.

Typical build-up speeds are 15 seconds per cycle for a full platform of approximately 100x50 mm and a layer thickness of 15 µ. Therewith, by a 100% use of the platform a build-up speed of 300 mm^3/min is realised.

The use of a slurry reduces safety precautions for using metal powders often seen with SLM processes. And although debinding and sintering processes are needed, there is no or hardly any component rework or deburring needed, as in SLM processes.

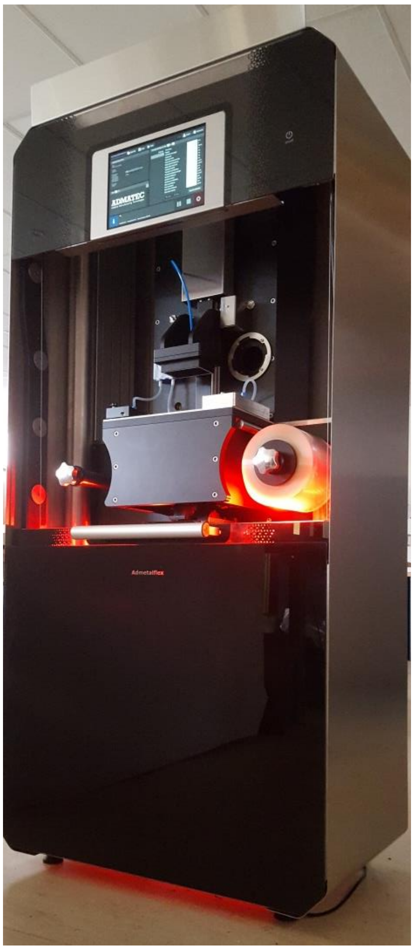


Figure 9: Admetalflex 130 (for printing metals).

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Conclusion

VAT stereo-lithography 3D printing, formerly successfully applied by Admatec / ECN to manufacture high performance ceramics, has now also been proven for printing of high density metals.

While the 3D form is realised by photo initiated curing, indentation depth of light in the slurry is critical for a reliable and economically feasible process. This can be solved by optimising the slurry compositions refractive index and particle size and by applying a printer capable of making thin layers with micron accuracy of the uncured slurry. Debinding and sintering of the end product is thereafter equal with existing MIM processing.

With this process, high quality components can be produced. These components can have an intricate design and are stress free with low surface roughness, as shown for 316L stainless steel.

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