High-throughput material development using selective laser melting and high power laser

Konstantin Vetter[1,a], Hannes Freiße[1,b] and Frank Vollertsen[1,2,c]

[1] BIAS - Bremer Institut für angewandte Strahltechnik GmbH, Klagenfurter Straße 5, 28359 Bremen, Germany

[2] University of Bremen, Bibliothekstr. 1, 28359 Bremen, Germany

[a]vetter@bias.de, [b]freisse@bias.de, [c]info-mbs@bias.de

Keywords: Alloy, Selective laser melting (SLM), Material development

Abstract. A novel approach for a higher efficiency in the discovery of new metallic alloys is based on a high-throughput method in order to provide materials with a precisely adapted requirements profile. An elementary part of this is the realization of reproducibly homogeneous alloy compositions. For this purpose, a process chain of two laser-based processes, selective laser melting (SLM) and high power laser, is used. Powdered stainless steel is applied on an unalloyed case hardening steel by SLM before base material and master alloy is remelted and mixed using a modulation form of consecutive overlaying circles of different sizes. In the determination of the homogeneity of the alloy element distribution within the melt pool, a homogeneous mixing of the main alloy elements chromium and nickel is achieved over the entire sample. It can also be shown that with increasing laser power, the chromium content in the sample decreases. The increasing thickness of pre-deposited layers leads from a certain thickness to a nearly linearly increasing chromium and nickel content.

Introduction

The steady growing demands on product properties and quality, as well as growing global competition require innovations in areas such as energy generation and transformation, mobility, infrastructure, and safety. Metallic materials, in particular, steels, are by far the world's most widely used construction material [1]. The development of new metallic construction materials, which are specifically tailored to the requirements, is an approach to realise innovative future technologies.

Conventional material development processes require many resources and extensive experimental investigations to determine the material properties. A basic understanding of the relationships is necessary to realise a target-oriented and model-based bottom-up solution. So far, models are missing due to many different demands placed in today`s world of materials. Hence, only a low number of experiments are possible, so that the procedures are mostly predictive and discovering completely new materials is made more difficult [2]. In this work, a novel and highly efficient method to discover new metallic materials is introduced.

Approach

A new method based on high-throughput was proposed in 2014 to enable a significantly higher efficiency in the discovery of new materials, or even new material classes [2]. The principles of high-throughput screening are shown in Fig. 1. Firstly, micro samples with a high-throughput must be generated. Secondly, thermal, mechanical or thermo-mechanical treatments are introduced in the microstructure of the samples. Now the materials are examined with respect to their characteristics, the so-called descriptors. Descriptors are measurable values which allow conclusions about the final material properties. Based on an evaluation function, the feedback from micro samples is used to produce samples for next generation. If a certain quality threshold is reached, a macro sample is generated for validation [3]. The transfer of the descriptors to macroscopic material properties is

carried out by scaling effects and requires only a few macro samples. This innovative approach provides target-oriented and resource-efficient alloy compositions as well as process chains for new metallic construction materials. These shall meet a requirement profile specified by the user regarding material properties such as flexural strength, toughness or machinability.

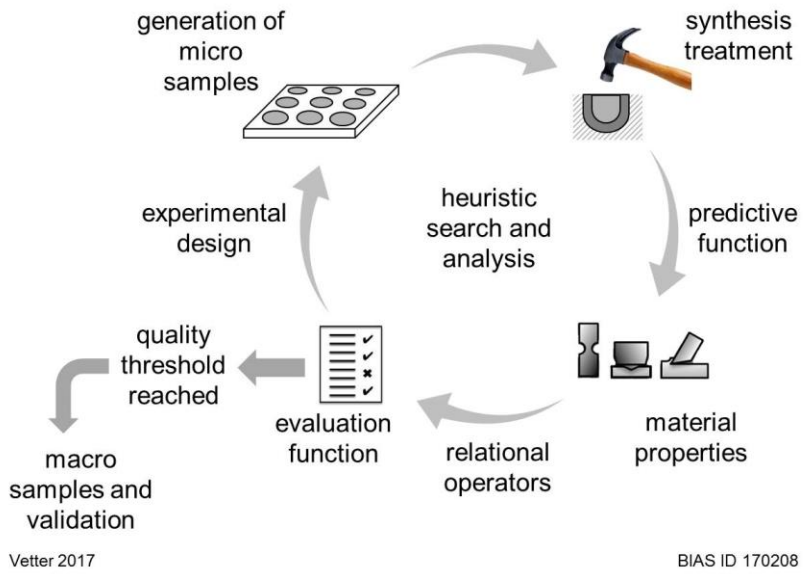


Figure 1: High-throughput screening for material development

One Part of this is to replace the conventional molding process and generate reproducibly, homogeneous alloy compositions. In order to realise a high-throughput process, research is carried out on a methodology based on the use of two laser-based processes in a new process chain. Fig. 2 illustrates the individual functions of both processes. First, alloy element layers are pre-deposited on the base material by SLM. In the next step, the alloy element layers are remelted and mixed with the base material by a scanner-guided laser deep alloying process.

To ensure high reproducibility, a defined melt volume and accordingly a defined alloy composition must be adjustable. For this reason, the penetration depth is to be determined directly in the process using low-coherent interferometry. This method has already been used in deep-penetration laser welding investigations. A close match of the measured results with the actual penetration depth was achieved [4]. In-situ detection of the emission spectrum of the process-induced metal vapour is carried out to determine the homogeneity of the material composition. It is assumed that the characteristic lines of the spectrum no longer change in a fixed area when a uniform dilution of the melt pool is achieved. This method has already been used to record changes of element parts during deep-penetration laser welding [5]. The beam modulation strategy directly influences the geometry of the melt volume and the uniformity of the dilution. Previous investigations demonstrated the influence of different laser beam movements on the distribution of particles in a melt pool during laser deep dispersing. Compared to pure linear feed movement, superimposed movement forms lead to a turbulent melt pool flow [6]. In this way, a homogeneous mixing of the pre-deposited alloy elements with the base material is achieved. Other studies analysed the distribution of hard particles during laser deep dispersing. They showed that the homogeneity of the melt volume is also influenced by additional material [7]. Studies on wire-based laser alloying also showed that circular oscillation is well suited for a homogeneous dilution [8].

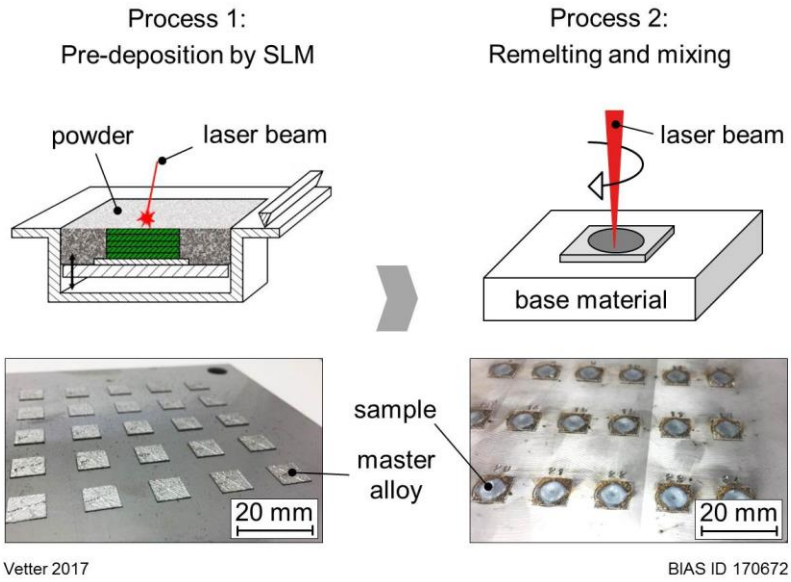


Figure 2: Process chain for the generation of deep-alloyed micro samples

Experimental Procedure

Materials. The unalloyed case hardening steel C15 acted as base material. Melting occurs at a temperature around 1510 °C. Powdered stainless steel X2CrNiMo17-12-2 was used as a master alloy for pre-depositing of alloy layers on the base material. The grain size was from 10 µm to 45 µm. The chemical composition is listed in Table 1. After pre-deposition of the master alloy by SLM, the pure layer was examined for the chromium and nickel content by means of energy-dispersive X-ray analysis (EDX). The arithmetical average of three measurements gives a chromium content of 14.8 % by weight and a nickel content of 10.2 % by weight. Thus the measured values differ by 10 % to 22 % compared to the values given by the manufacturer for pure powder as indicated in Table 1.

Table 1: Chemical composition of X2CrNiMo17-12-2

Element	**C**	**Si**	**Mn**	**Ni**	**Cr**	**Mo**
Weight percent (%)	0.012	0.56	0.31	13.1	16.5	2.27

Experimental setup. The pre-deposition of the master alloy was carried out by using a ReaLizer SLM 250 with a wavelength of 1070 nm. The single layers of the total layer were applied with a thickness of 50 µm. For the investigations of the influence of the laser power and the modulation speed during remelting and mixing, layers with a total thickness of 0.2 mm were generated by SLM with a laser power of 100 W and a scanning speed of 120 mm/s. Investigation on the influence of different layer thickness was carried out on layers with a total layer thickness between 0.1 mm and 0.5 mm by using a scanning speed of 80 mm/s. The layers have a square shape with a side length of 10 mm and were pre-deposited on a base material plate with the dimensions 150 mm x 150 mm x 18 mm.

The remelting and mixing of the element layers with the base material were carried out with a disk laser Trumpf TruDisk12002 with a wavelength of 1030 nm and a maximum power of 12 kW using a 3D-Scanner-Optic Trumpf PFO 3D with an f-theta lens and a focal length of 450 mm. Fig. 3 a) illustrates the experimental setup. The spot diameter of 650 µm was kept constant. Argon

shielding gas was provided at a rate of 30 standard liters per minute laterally at an angle of 90° using a nozzle with a diameter of 7 mm. The laser beam was modulated circularly by the scanner optics applying a form of 14 consecutive overlaying circles of different sizes and a total modulation path of 115 mm as shown in Fig. 3 b).

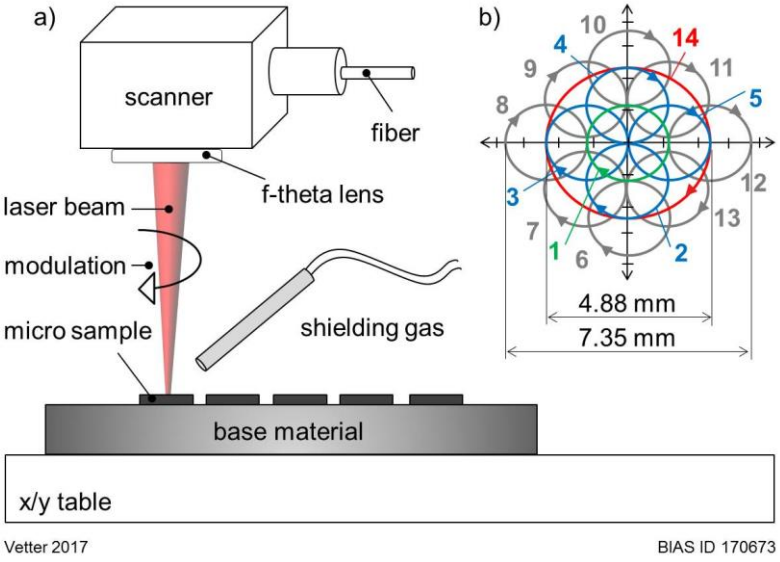
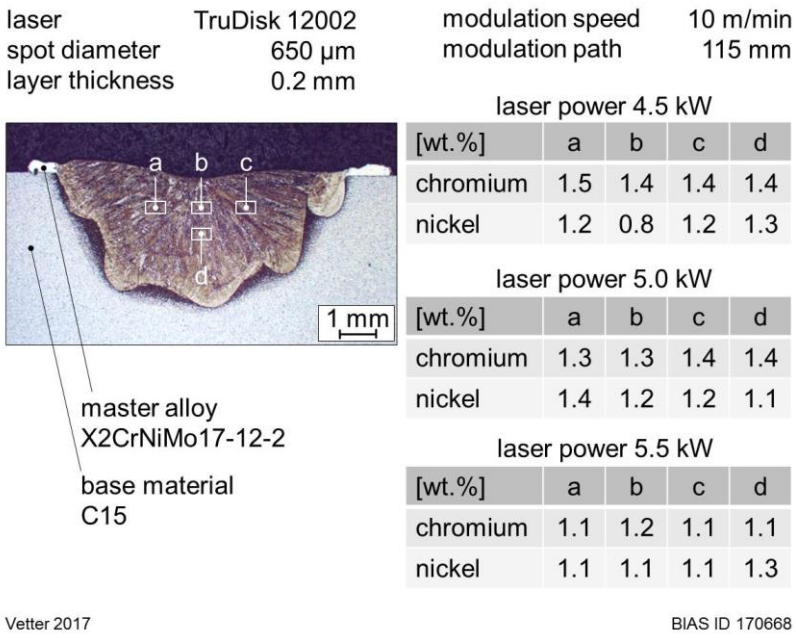


Figure 3: a) Principle of the laser deep alloying process, b) Modulation form with a pathlength of 115 mm for remelting and mixing the pre-deposited layer with the base material

Results and discussion

Influence of laser power. For the investigation of the influence of the laser power on the generation of deep alloyed micro samples, the laser power was varied in steps of 500 W within a range from 4.5 kW to 5.5 kW. In order to determine the homogeneity of the alloy element distribution within the melt pool, micro samples were examined by EDX analysis. The analysis results show the concentration of the elements chromium and nickel distributed in the areas of the micro sample defined by the rectangles as shown in Fig. 4. In the upper zone of the microstructure, there is constant chromium content about 1 % by weight. The nickel content is also constant, even though at a lower concentration. The element content in the middle zone of the micro sample is as high as in the upper region. This indicates homogeneous mixing of the entire sample. The homogeneity of the element distribution is not influenced by the laser power. The influence of the laser power on the penetration depth is still under investigation. However, the expected trend of decreasing element content with increasing laser power can be shown for chromium. This can be explained by the larger melt pool, whereby the alloy elements are spread over more base material.

Cross sections show that the microstructure does not change for all samples that are produced with a laser power in this range. The cooling takes place from the outside to the inside of the sample, so that a directed microstructure occurs.



laser power 4.5 kW

[wt.%]	a	b	c	d
chromium	1.5	1.4	1.4	1.4
nickel	1.2	0.8	1.2	1.3

laser power 5.0 kW

[wt.%]	a	b	c	d
chromium	1.3	1.3	1.4	1.4
nickel	1.4	1.2	1.2	1.1

laser power 5.5 kW

[wt.%]	a	b	c	d
chromium	1.1	1.2	1.1	1.1
nickel	1.1	1.1	1.1	1.3

Figure 4: EDX analysis of samples produced with different laser power. Chromium and nickel content in weight percent. EDX analysis windows are defined by the rectangles

Influence of modulation speed. The energy applied to the melt pool not only depends on the laser power but also on the modulation speed of the scanner. For the investigation of the influence of the modulation speed on the penetration depth and the microstructure of deep alloyed micro samples, the modulation speed was varied in three steps from 5 m/min to 10 m/min. The penetration depth was measured from the original surface to the deepest penetration of the melt pool like shown in Fig. 5. The values in the diagram are an arithmetic average of three measurements with the standard deviation. With a laser power of 4 kW and a modulation speed of 10 m/min, the penetration depth is 3 mm. Using the same laser power at half modulation speed of 5 m/min the penetration depth is increased by 43 % to 4.3 mm.

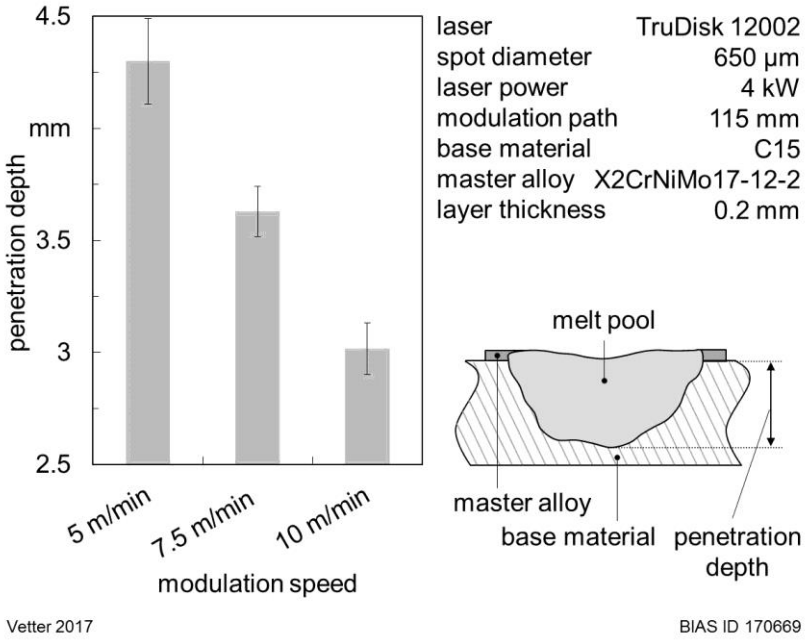


Figure 5: Penetration depth depending on the modulation speed

The comparison of the cross sections in Fig. 6 illustrates the enlargement of the melt pool at a lower modulation rate. The cross section of the sample generated with a modulation speed of 5 m/min shows that the longer exposure time of the laser does not only affect the penetration depth. Rather, the entire geometry of the sample is influenced. The melt pool geometry is formed from a semicircular shape at a high modulation speed to a square shape at a low modulation speed.

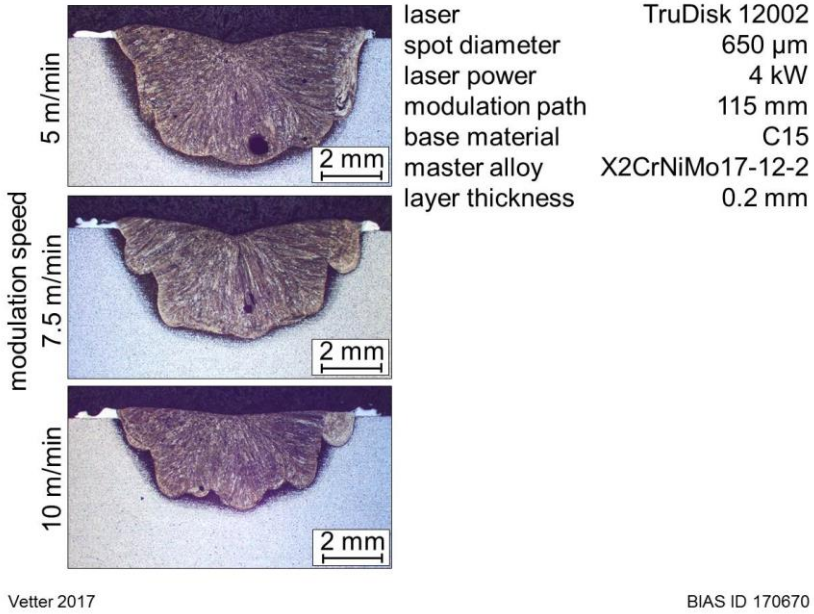


Figure 6: Cross sections of samples produced with different modulation speed

Influence of layer thickness. In Fig. 7 the influence of the layer thickness of the master alloy on the chromium and nickel content in weight percent is shown. The values in the diagram for the alloy content are an arithmetic average of three EDX measurements. At a layer thickness of 0.1 mm, the chromium content is 0.5 % by weight and the nickel content is 0.4 % by weight. At a layer thickness of 0.5 mm, the chromium content is 3.6 % by weight and the nickel content is 2.8 % by weight. It can be seen, that the alloying ratio of chromium to nickel is the same at all layer thicknesses, however with a low scatter. The alloying ratios are slightly below the alloying ratio of 3:2 of the master alloy applied by SLM.

With an increasing layer thickness the chromium and nickel contents also both increase. Above a certain layer thickness, the chromium and nickel contents increase in a nearly linearly increasing course. The increase of the alloying elements is then almost proportional to the increase of the layer thickness.

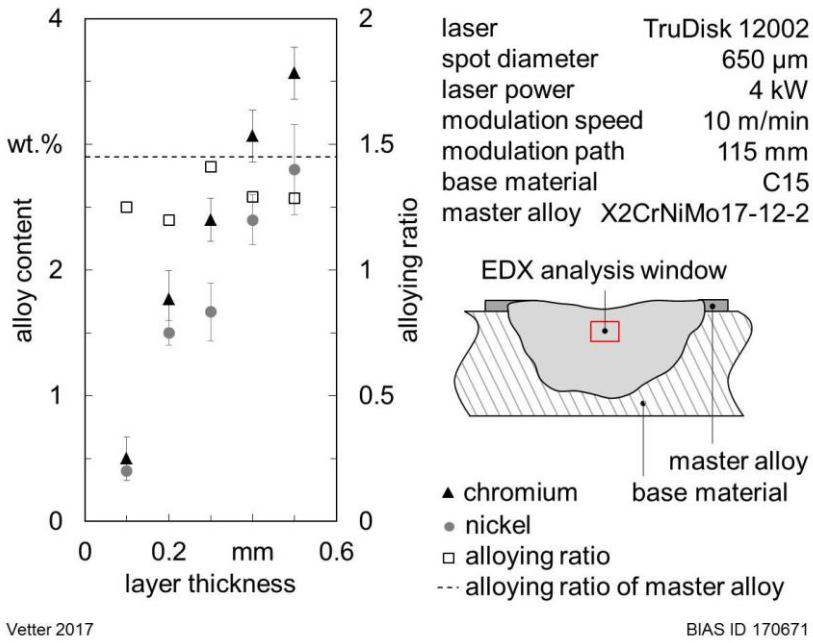


Figure 7: EDX analysis of samples produced with different layer thickness. Chromium and nickel content in weight percent as well as the alloying ratio of chromium and nickel as a function of increasing amount of master alloy. EDX analysis window is defined by the rectangle

Conclusions

The application of different layer thicknesses of the master alloy leads from a certain layer thickness to an almost linear increase of alloy content in the micro sample. Therefore, the laser deep alloying process using pre-deposited element layers combined with beam modulation is a suitable method to adjust the alloy content specifically and achieve alloys with a homogeneous dilution of alloy elements.

Acknowledgement

Financial support of subproject U02 `Laser deep alloying for the realization of finely graded alloys` of the Collaborative Research Center SFB1232 by the German Research Foundation (DFG) is gratefully acknowledged.

References

[1] The Swedish metals-producing Industry`s associations: The Swedish Steel producers Association (Jernkontoret), National action for metallic materials – A Strategic Research And Innovation Agenda, (2013) 8.

[2] L. Mädler, Is High-throughput screening for structural material/metals possible? in: Proceedings of the 4th International Conference on Nanomanufacturing – nanoMan, Bremen, Germany, 8-10 July (2014).

[3] R. Drechsler, S. Eggersglüß, N. Ellendt, S. Huhn, L. Mädler, Exploring Superior Structural Materials Using Multi-Objective Optimization and Formal Techniques, 6th IEEE International Symposium on Embedded Computing and System Design – ISED, Patna, India, 15-17 December (2016).

[4] P.J.L. Webster, L.G. Wright, Y. Ji, C.M. Galbraight, A.W. Kinross, C. Van Vlack, J.M. Fraser, Automatic laser welding and milling with in situ inline coherent imaging, in: Optic letters 39 (2014) 6217-6220.

[5] S. Huber, In-situ-Legierungsbestimmung beim Laserstrahlschweißen, Dissertation, Technische Universität München, (2014) 129-130.

[6] F. Vollertsen, K. Partes, G. Habedank, T. Seefeld, Deep penetration dispersing of aluminum with TiB_2 using a single mode fiber laser, in: Production Engineering – Research and Development 2, (2008) 27-32.

[7] J. Volpp, A. Zingraf, F. Vollertsen, Impact of process parameters on particle distribution and wear resistance during laser deep alloying processes, in: Proceedings of the International Congress on Laser Advanced Materials Processing – LAMP, Japan Laser Processing Society (2015).

[8] K. Hofmann, F. Hugger, M. Holzer, S. Roth, M. Schmidt, Drahtbasiertes Laserstrahllegieren zur Erhöhung der Verschleißbeständigkeit von Presshärtewerkzeugen für die Warmblechumformung, in: Tagungsband zum 10. Erlanger Workshop Warmblechumformung, Meisenbach Bamberg (2015) 151-163.