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Procedia CIRP 111 (2022) 111–114

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12th CIRP Conference on Photonic Technologies [LANE 2022], 4-8 September 2022, Fürth, Germany

Advances in powder bed based Additive Manufacturing of metal-glass-hybrid-components

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Abstract

As powder bed based additive manufacturing (AM) is gaining more traction in the research and industry, significant efforts are made to expand the capability of AM in combining different materials. Conventionally, multiple materials, sealants and processes are necessary in order to join metal and glasses in assemblies. The development of a single-step process for vacuum-tight and strong bonding between glass and metal could drastically reduce the part count and assembly cost.

This paper investigates the feasibility of joining the iron-nickel-cobalt alloy Kovar with borosilicate glass. The powder bed fusion by laser beam is used to melt Kovar structures directly onto glass substrates. Suitable process parameter for an iron-silicon-oxide material-bonding layer of 3 µm thickness were developed using design of experiments. With an energy input of 333 J/mm^3 for the initial layer and 60 J/mm^3 for the bulk structure, tensile testing of the metal-glass-connection revealed bonding forces up to 0.5 MPa.

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1. Introduction

Due to the research and attention that metal additive manufacturing has received since its development, it is now possible to use its disruptive properties in an increasing number of application areas in the industry. This work focuses on the joining of metal and glass materials. An area, which extends from joining parts for applications in medicine, electronics, aviation or power engineering. Through the similar thermal expansion coefficient, glass-to-metal-seals (GTMS) have been designed with an Iron-Nickel-Cobalt alloy (Kovar) to join with borosilicate glass [1, 2]. A material bond could be proven between the connection of iron, silicon and oxygen [3-5]. The chemical composition and the microstructure of the bond was analyzed and Fe_2SiO_4 was stated to be the interface material [4, 5]. A crucial factor in the creation of hermetic GTMS is also the wetting behavior of the Kovar on the glass, which is improved by a pre oxidation of the metal and a tailored heating and cooling procedures during the joining process [4, 6, 7]. To the knowledge of the authors, no attempts have been made to create GTMS with the powder bed fusion by laser beam (PBF-LB). This work lays the foundation of transferring the state of the art to the PBF-LB. Therefore, the possibility of a material bond between Kovar and borosilicate glass in a state of the art industrial PBF-LB system is investigated, analyzing the interface and tensile testing the connection of metal and glass.

2212-8271
10.1016/j.procir.2022.08.066

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2. Materials and methods

2.1. Machine set up and materials

The machine used in this investigation is the PBF-LB laser melting system TruPrint 1000 by Trumpf (Ditzingen, Germany). The laser is a continuous wave ytterbium fiber laser with a wavelength of 1070 µm, an output power of 200 W and a focus diameter of 30 µm. Sandvik (Sandviken, Sweden) provides the Kovar powder. The material consists of 54 % Iron, 29 % Nickel and 17 % Cobalt and is gas atomized to a particle size distribution of 15 to 53 µm (Fig. 1). The borosilicate glass substrates are acquired from Schott (Mainz, Germany). The substrates were laser cut to an edge length of 4 by 12 by 38 mm.

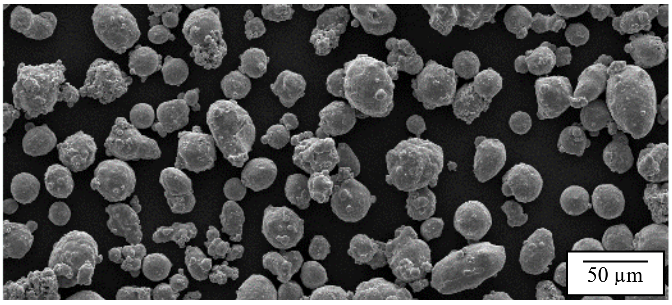

Fig. 1. Kovar powder

In order to melt the metal powder onto the glass surface, two substrates are glued diagonally to a Kovar substrate plate in the build chamber of the PBF-LB machine. The resulting void around the glass is filled with powder to enable a standard PBF building process. After the building process, the specimens are embedded in epoxy resin. Cross sections of the embedded specimens are made by grinding and polishing with a Tegramin-30 by Stuers (Ballerup, Denmark). To analyze the interface between the metal structures and the substrates microscopic images are made with the laser scanning microscope VK-X1000 by Keyence (Neu-Isenburg, Germany). SEM Images as well as energy dispersive X-ray spectroscopy (EDX) for spot-measurements, line scans and element mappings are made with the Quanta 400 FEG by FEI (Hillsboro, Oregon, USA). Tensile testing was accomplished with the Unimat Plus 052-5kn by Erichsen (Hemer, Germany).

2.2. Experimental set up and methods

The proof of a material bond between PBFed Kovar and glass substrates must be provided as a base for further investigation. To achieve this connection, the first part of this research examines the interface by applying layers of Kovar powder on the borosilicate glass and melting the powder with a laser beam. The first experiment investigates the energy input and the number of applied Kovar layers to create a thin homogenous metal deposition on top of the glass surface. The laser beam is exposing a round surface with a diameter of 6 mm, a hatching distance of 30 µm and a powder layer height of 20 µm. For maximizing the expressive value while minimizing the extent of this investigation, methods from the Design of Experiments (DoE) are used. Therefore, a central composite design (CCD) is applied for the first experiment. The parameters are shown in Tab. 1.

Table 1. Process parameters of the CCD design for the metal-glass-interface

#	Laser power [W]	Scanning speed [mm/s]	Initial deposition height [µm]	Extended deposition height [µm]
1	25	400	40	100 / 200
2	35	400	40	100 / 200
3	25	800	40	100 / 200
4	35	800	40	100 / 200
5	25	400	80	100 / 200
6	35	400	80	100 / 200
7	25	800	80	100 / 200
8	35	800	80	100 / 200
9	20	600	60	100 / 200
10	40	600	60	100 / 200
11	30	200	60	100 / 200
12	30	1000	60	100 / 200
13	30	600	20	100 / 200
14	30	600	100	100 / 200
15	30	600	60	100 / 200

After the laser melting process, the substrates are extracted and embedded into epoxy resin. Microscopic images are taken to analyze cross sections of the Kovar-glass-interface. The second experiment was used to apply the knowledge of the first experiment to extend the height of the metal deposition. With the same laser parameters and geometries, the experiment built cylinders of 100 and 200 µm height onto the glass surface. The laser parameters are given in Tab. 1.

If a material bond can be detected, the second part of this work regards the fabrication of tensile specimens onto the interfacing layers to evaluate the strength of the connection. The tensile specimens are specially designed to have a 6 mm cylindrical contact patch with the glass substrate and a teardrop shaped gap to connect to the tensile testing machine. In order to get further insights of the laser parameters, a full factorial design is applied for the interface. The laser power is investigated between 40 to 70 W in 5 W increments and the scanning speed ranges from 300 to 1000 mm/s in 100 mm/s steps. The geometry of the specimen is built with a set of preliminary investigated parameters (13 W, 340 mm/s). This parameter set leads to specimen with a high porosity but low temperature induced curling, therefore minimizing shear stress on the metal-glass-interface in the raking process. It was preliminary shown that the porous geometry does not limit the tensile strength. Fig. 2 shows the geometry of the tensile specimen, the substrates and the placements of the specimens.

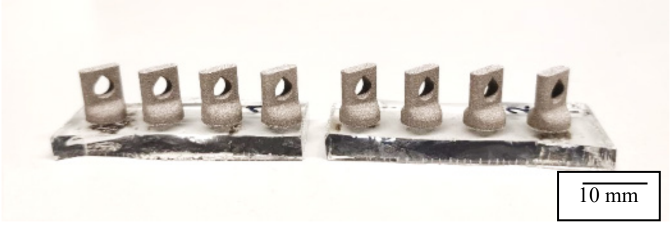

Fig. 2. Kovar tensile specimen on borosilicate substrate

3. Experimental Results

3.1. Results of the 1st part – material bond

The first investigation was set up to fabricate and analyze an interface between the metal powder and the borosilicate substrate. One to five layers of Kovar melt were initially deposited onto the glass substrate with the laser parameters shown in Tab. 1. This investigation shows that, due to the machine tolerances, a homogenous powder application over the entire glass substrate surface is only guaranteed from the fourth layer onwards. The Kovar melt shows a strong balling behavior on top of the glass substrate. This is depicted in Fig. 3. With high probability, the melted Kovar has a strong cohesive force, resulting in a large contact angle. Due to the thermal energy of the particle in the molten state (Temperature above 1449°C) and in a solidified state over the melting point of the borosilicate surface (820°C) a temperature transfer into the glass locally melts the surface. Fig. 3 illustrates the capillary rise of the liquid borosilicate, which leads to an enclosure of the Kovar sphere in the glass substrate.

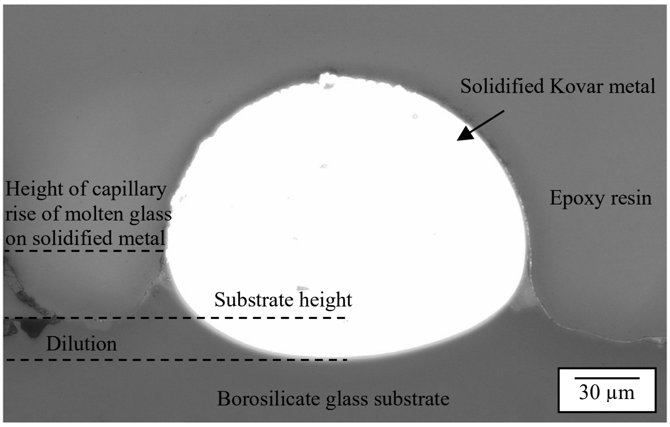

Fig. 3. Results of the initial metal deposition – Kovar melt on substrate

A second investigation is set up to extent and homogenize the deposited metal on top of the substrate. Therefore, the number of applied Kovar layers have been increased. This experiment showes that, especially at high energy inputs, a temperature induced curling of the deposited metal can occur after six to eight completed process cycles. The connection between the molten metal and the glass is strong enough to tear off a thin layer of glass and lift it with the curvature of the metal. These failures can lead to an extraction to the build structure in the powder raking process. This cracking of the glass is depicted in Fig. 4. The figure shows one of the specimen build with a high energy input and ten layers of material (Laser power = 30 W, scanning speed = 200 mm/s). This specimen was not removed by the raking process and the connective mechanisms can be analyzed. The overview is taken with a laser microscope and detailed images (Fig 4 A) and two EDX-line-scans (Fig. 4 B & C) of the connection are recorded in the SEM. The overview shows that there is still a balling behavior and an unevenness of the melted Kovar, yet some connected particles and areas can be observed. There is a curvature in the Kovar depostion, which lead to the cracks in the glass substrate. This suggests that the connection between the metal and the glass was stronger than the glass, leading to the curved cracks under the Kovar particles and moreover, the laser radiation and the temperature input weakens the glass substrate. The Fig. 3 shows a SEM images with a backscatter detector highlighting the contrast between different materials. There are three phases visible: A Kovar, a borosilicate and an boundary phase. To analyze this phases, line-scans were carried out in two different areas. Fig. 4 B shows a thin layer, which appears between particles and the glass. Fig. 4 C shows a board area of this connecting phase, which appears in some areas around irregular solidified Kovar melt.

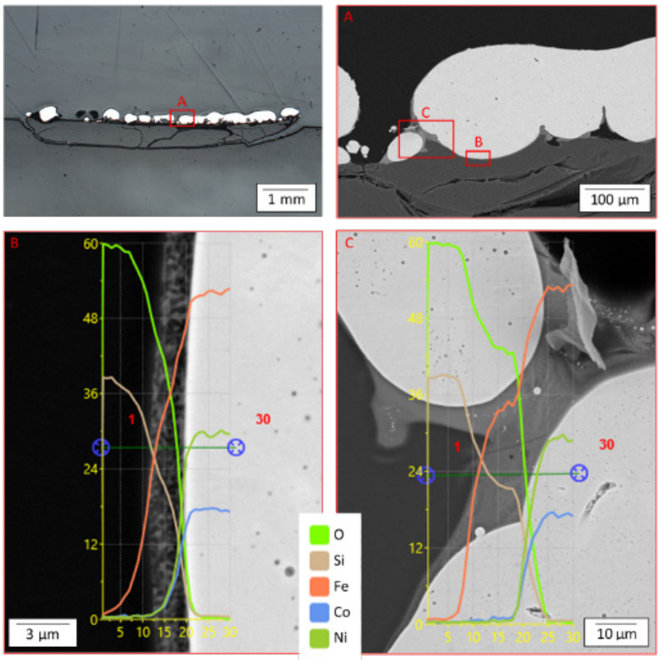

Fig. 4. Results of the extended metal deposition: Top-left) Overview laser scanning microscope; A) SEM image of area of interest; B) EDX-line-scan in connective layer; C) EDX-line-scan in irregular area

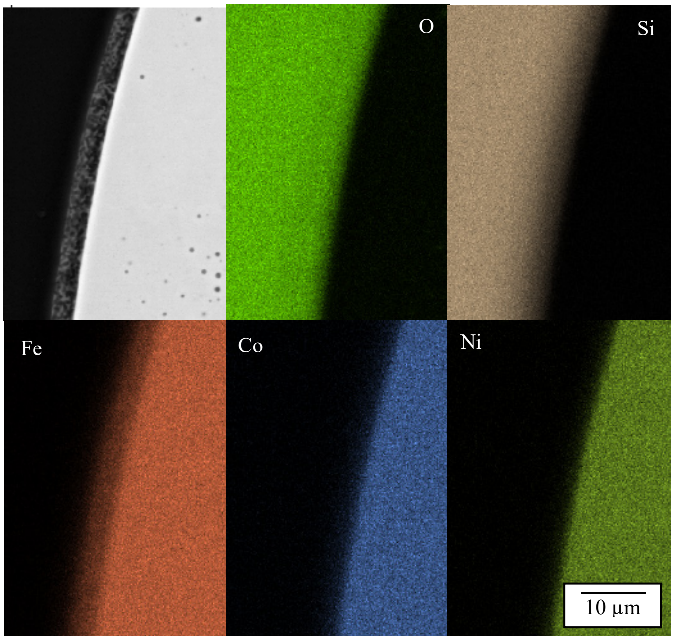

Fig. 5. Mapping of elements of fig. 4 C.

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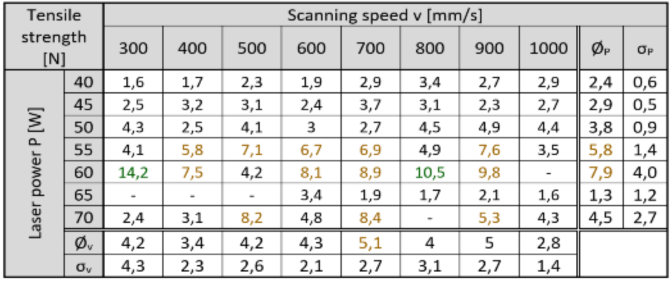
The line-scan consists of 30 points of measurement detecting the chemical composition in mass percentage. At the start (left) only the silicon and oxygen of the borosilicate and at the end the iron, nickel and cobalt of the Kovar could be detected. In the boundary area is a diffusion of iron into the borosilicate. To further analyze the interface a mapping of elements of the area in Fig. 4 B has been made. This is shown in Fig. 5. It can be seen that the silicon has a gradient towards the interface and the iron has a visible step in intensity, whereas cobalt and nickel seem to have a clear boarder. This strengthens the observations of [3-5] and shows the possibility of an iron-silicon-oyxgen or even a Fe_2SiO_4 interface. Therefore, the proof of a material bond is provided.

3.2. Results of the 2nd part – tensile testing

To evaluate the connective force and the limitation of the connection tensile tests were carried out. Therefore, five initial layers with high energy inputs are built, while the rest of the specimens will be fabricated with a lower energy input. The results of the tensile tests are given in the figure 6. The value of the tensile force F_{max} (ultimate strength) is stated as well as the mean values and standard deviations for the factor. Values above 5 N are highlighted yellow and above 10 N green. In tensile testing the connection showed no plastic deformation



Tensile strength [N]		Scanning speed v [mm/s]									
		300	400	500	600	700	800	900	1000	$Ø_P$	σ_P
Laser power P [W]	40	1,6	1,7	2,3	1,9	2,9	3,4	2,7	2,9	2,4	0,6
	45	2,5	3,2	3,1	2,4	3,7	3,1	2,3	2,7	2,9	0,5
	50	4,3	2,5	4,1	3	2,7	4,5	4,9	4,4	3,8	0,9
	55	4,1	5,8	7,1	6,7	6,9	4,9	7,6	3,5	5,8	1,4
	60	14,2	7,5	4,2	8,1	8,9	10,5	9,8	-	7,9	4,0
	65	-	-	-	3,4	1,9	1,7	2,1	1,6	1,3	1,2
	70	2,4	3,1	8,2	4,8	8,4	-	5,3	4,3	4,5	2,7
	$Ø_v$	4,2	3,4	4,2	4,3	5,1	4	5	2,8		
	σ_v	4,3	2,3	2,6	2,1	2,7	3,1	2,7	1,4		

Fig. 6. Results of the tensile testing

The results indicate that the laser power has a significate influence while the scanning speed remains insignificant. The connective force seems to increase up to 60 W then dropping abrupt at 65 W. A maximum force of 14.2 N (0.5 MPa) was measured at 60 W laser power and 300 mm/s scanning speed.

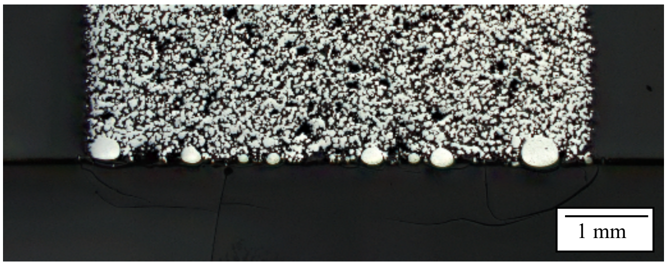


Fig. 7. Cross section of a tensile specimen

Fig. 7 shows a cross section of the lower part of a tensile specimen with 50 W and 600 mm/s as parameters. It can be seen that similar to the previous experiments, the balling still seems to dominate the interface and the connection between metal and glass. Some cracks are already forming in the glass, explaining the sudden drop in tensile strength at 65 W.

The tensile specimen is very porous yet strong enough to not be the weakest link. The tensile force seems to be transferred through the big spheres of the balling, which are attached to the glass. To confirm the best parameter and showcase the findings of this investigation, a demonstrator part of fabricated (Fig. 8.)

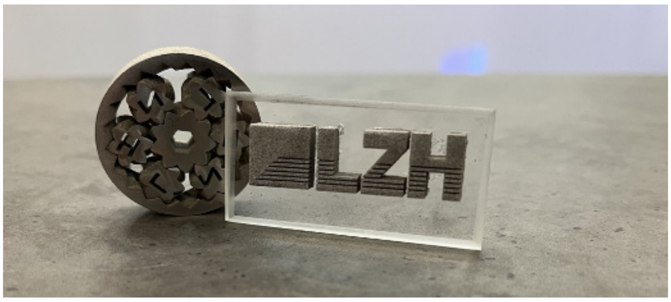


Fig. 8. Kovar demonstrator connected to borosilicate glass

4. Conclusion

This paper investigates metal-to-glass joining in the PBF-LB. The following findings could be made:

- With Kovar as a powder material and borosilicate glass substrate a material bond could be proven
- EDX-line-scans and elemental mappings show that the connection consist out of iron, silicon and oxygen.
- The nickel and cobald of the Kovar do not seem involved in the interface
- Kovar shows a balling tendency, but the glass seems to adhere to the solidified spheres in a liquid state
- Tensile testing reveled bonding forces up to 0.5 MPa

With this knowledge, the authors recommend investigating into further powder materials like Invar (64% Fe & 36 % Ni) for lower thermal expansion and higher iron content. In addition, a change in glass substrate like sapphire glass could lead to improved tensile strength due to less cracking. Without changing the materials further improvements in the connection would be expected when:

- Build chamber preheating and peroxidation for better wetting of the substrates (less balling of Kovar) and more even interface
- Increased laser spot for less laser intensity to reduce thermal impact on the substrate leading to less cracks
- Mechanical treatment of the glass substrate (e.g. milling) to increase laser absorption into the surface

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