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A Comparative Study on Different Joining Techniques For PM-HIP Evacuation Tubes

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Abstract

The main purpose of evacuation tubes in powder metallurgically-based (PM) hot isostatic pressing (HIP) lies in the evacuation and outgassing of the capsules. Conventional HIP capsules are made of sheet metal which exhibits good weldability and therefore, it is easy to join the evacuation tube. With the emerging technologies of Additive Manufacturing (AM) it is now possible to design more complex capsules for HIP. Additionally, wear-resistant, carbide rich steels can be utilized. Yet, these materials are known to be difficult to weld. This study compares two different approaches to bond an AISI 304L evacuation tube to a HIP capsule made by Electron Beam Melting (EBM) from high carbon tool steel AISI A11. Capsules were bonded by TIG welding as well as brazing with conventional and a customized filler material based on thermodynamic calculations. Subsequent consolidation was performed by HIP, microstructural analysis and argon-measurements revealed the feasibility and limits of all three approaches for gas-tight joints.

Introduction

Hot Isostatic Pressing (HIP) is a well-established process for the consolidation of metallic powders to solid material. It is among one of the most reliable forming processes for the production of near-net-shape parts in demanding industries, such as aerospace, automotive, or oil and gas [1]. The powder is filled into a thin-walled capsule using one or several filling tubes. To achieve a preferably high packing density, the filling process often takes place under constant vibration [2]. The bulk powder within the capsule is afterwards degassed through the evacuation tubes via a vacuum pump and kept under vacuum for several hours. While still under vacuum, the capsule can be closed by forging and welding of the evacuation tubes. The densification of the encapsuled and degassed powder compact within the HIP vessel under elevated temperature and high pressure [3,4] is the last step before removing the capsule by sawing, turning, or milling to obtain the finished part. The whole HIP process chain is illustrated in the following ***Figure*** .

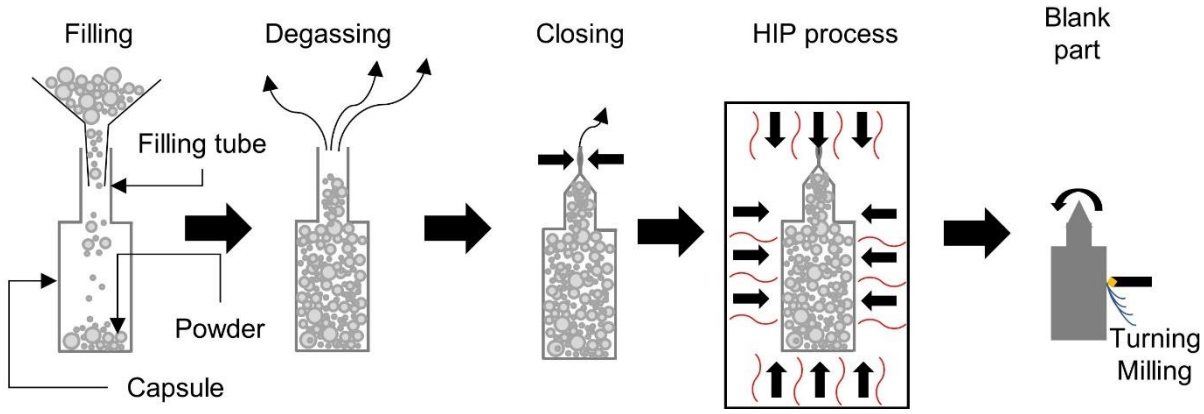


Figure 1. The process chain of powder metallurgical HIP and the role of the evacuation tube

Conventional capsules for powder metallurgical HIP (PM HIP) are welded from sheet metal [2], in case of Fe- or Ni-alloys utilizing austenitic grades such as AISI 304L or mild steel. The geometries of the finished parts are limited in complexity due to the manual fabrication process of the capsule. With the emerging technology of additive manufacturing (AM) it is possible to build far more complex geometries with few restrictions [5,6]. Therefore, AM can complement and improve the PM-HIP process chain by lifting geometric restrictions in capsule manufacturing [7–9]. Additionally, the range of available materials

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for the capsules can be broadened beyond the austenitic grades [10]. The combined AM+HIP approach opens the possibility of manufacturing wear-resistant composite components, where a wear-resistant shell, which serves as a HIP capsule is fabricated by AM and filled with a different powder material. The capsule remains on the finished part as a wear-resistant layer. Many wear-resistant materials exhibit poor weldability and forgeability due to their increased carbon content and low plastic forming potential [11,12]. Therefore, a designated and forgeable evacuation tube is necessary in order to close the capsule prior to the PM-HIP process.

An alternative joining method for materials with reduced weldability for high-temperature applications, such as the PM-HIP process, is diffusion brazing, also referred to as Transient-Liquid-Phase (TLP) bonding [13]. The main principle involved in TLP bonding is the diffusion of constituents from the interlayer filler material into the joining partners. Melting point depressing elements, such as silicon or boron, are alloyed with nickel- or iron-based material to form a near eutectic mixture. This mixture represents the filler material and is applied onto the joining partners with a defined gap thickness. Upon reaching the brazing temperature, the filler material undergoes a phase transition into the liquid state and wets the interface. The melting point depressants then start to diffuse into the joining partners at the isothermal brazing temperature, leading to a local change in chemical composition in the filler material and the top layer of the joining partners. Once depressants have diffused into the base material, the solidus temperature starts to rise and the filler material undergoes an isothermal solidification. Ultimately, no more liquid phase will be present and the joining process is completed [14–17]. As a result, the bond has a higher re-melting temperature after diffusion brazing than the original braze filler.

Materials and Methods

In this study, wear-resistant HIP capsules were fabricated using Electron Beam Powder Bed Fusion (PBF-EB) from the vanadium- and carbon-rich tool steel AISI A11 (X245VCrMo10-5-1) at the Fraunhofer Institute for Manufacturing Technology and Advanced Materials in Dresden, Germany. An Arcam A2X PBF-EB machine was used [10]. The capsule design for the experiments is depicted in Figure 1.

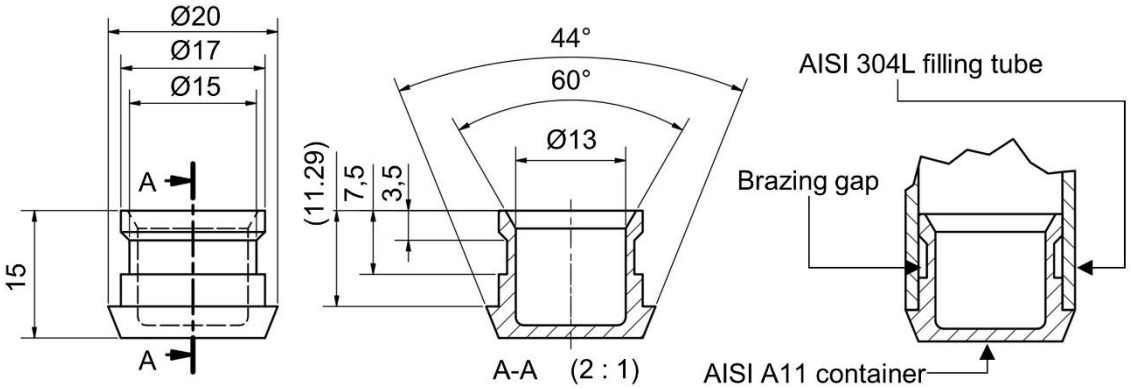


Figure 1. Capsule design for the joining experiments, produced from AISI A11 tool steel by PBF-EB

For the filling- and evacuation tube, the austenitic steel AISI 304L (X2CrNi19-11) was chosen due to its excellent forgeability. The capsules were filled with hot-working tool steel AISI L6 (56NiCrMoV7) powder. The combination of A11 and L6 steels for the components was chosen based on similar heat treatment conditions, leading to a maximum hardness of the wear-resistant capsule and high toughness of the powder. The chemical composition of the capsule material as well as the powder and evacuation tube is given in Table 1.

Table 1: Chemical composition of the HIP capsule, evacuation tube and powder filling in weight percent [wt.%]

Material	Part	C	Si	Mn	Cr	Mo	V	Ni	Fe
AISI A11	HIP capsule	2.4 - 2.5	0.75 - 1.10	0.35 - 0.60	4.75 - 5.75	1.10 - 1.50	9.25 - 10.25	---	Bal.
AISI L6	Powder	0.50 - 0.60	0.10 - 0.40	0.60 - 0.90	0.80 - 1.20	0.35 - 0.55	0.05 - 0.15	1.5 - 1.8	Bal.
AISI 304L	Evacuation Tube	< 0.03	< 1.0	< 2.0	18.0 - 20.0	-	-	8.0 - 12.0	Bal.

Two different joining processes were analyzed to bond the HIP capsule and the evacuation tube. In a first step, manual Tungsten Inert Gas (TIG) welding with special precautions in temperature control

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during the welding and subsequent cooling was investigated. Five joints were investigated and the joining zone was preheated to 700 °C before the welding process to prevent cracking. A welding current of 60 A was set and two passes were necessary to reach a sufficient penetration depth. AWS A5.28 ER80S-A1 (the chemical composition is given in Table 2) was selected as suitable filler material and the cooling was conducted in agglomerated silica submerged arc welding powder to prevent rapid cooling. Additionally, TLP bonding was investigated for its feasibility in joining PM-HIP evacuation tubes to capsules. Conventional TLP bonding was conducted on five joints in a vacuum furnace at a brazing temperature of 1190 °C and a dwell time of 2.5 h, using AWS A5.8 BNi-5 nickel-base filler material, see Table 2. This filler material was chosen based on its high melting range of 1080 °C - 1135 °C, which is close to the HIP temperature of 1150 °C. A second TLP bonding process was developed using inductive heating under argon protective atmosphere with a brazing temperature of 1130 °C and a dwell time of 30 minutes. In total, five inductive TLP joints were investigated. The inductive TLP process, as well as the capsule/evacuation tube assembly, are depicted in Figure 2 a) and b).

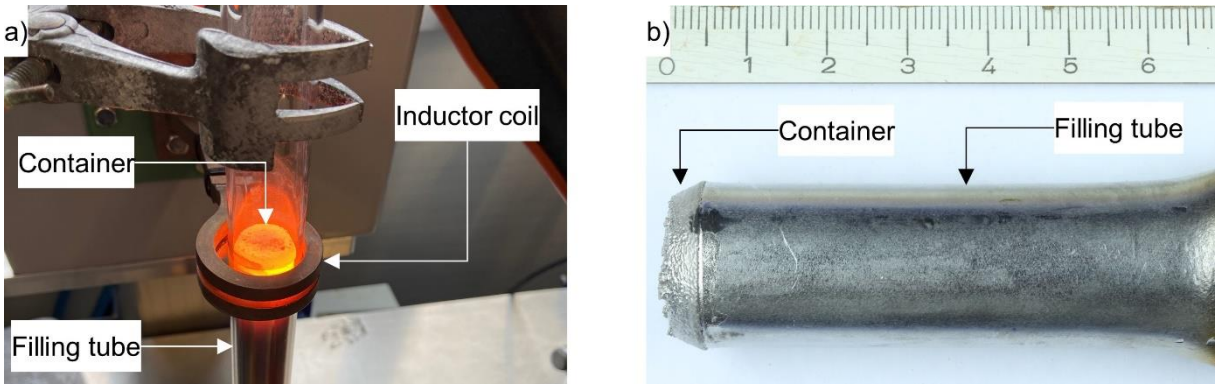


Figure 2. a) Inductive TLP process b) HIP capsule and evacuation tube assembly

The filler material was based on the AISI A11 capsule material and mixed with silicon as the melting point depressant in a near eutectic mixture. Therefore, 3.5 wt.% silicon was added and thickened with the binder Innobraze B2. The chemical composition and assessment of the eutectic point were chosen based on thermodynamic equilibrium calculations, using Thermo-Calc [18] with the TCFE9 database. The eutectic AISI A11-Silicon phase diagram is depicted in Figure 6 a). The brazing temperature, as well as the dwell time, were estimated by diffusion calculations with the Thermo-Calc DICTRA module. The chemical composition of both TLP bonding and the welding filler material is listed in Table 2.

Table 2: chemical composition of the welding and TLP bonding filler materials in weight percent [wt.%]

Material	Process	C	Si	Mn	Cr	Mo	V	Ni	Fe
AWS A5.28 ER80S-A1	Welding	< 0.1	0.6	1.1	---	0.5	---	---	Bal.
AWS A5.8 BNi-5	TLP Bonding furnace	< 0.06	9.7 - 10.5	---	18.5 - 19.5	---	---	Bal.	---
AISI A11 + 3.5 wt.% Si	TLP Bonding inductive	2.4	4.4	0.5	5.1	0.7	9.4	---	Bal.

Subsequent consolidation by HIP was conducted under Argon and the HIP parameters were set to a temperature of 1150 °C, a pressure of 100 MPa, and a dwell time of 240 minutes. The temperature and pressure were applied simultaneously for an efficient HIP process, although the AISI A11 capsule is susceptible to cracking at lower temperatures, due to its brittleness. Successful consolidation of the powder and therefore, a gas-tight bond between the evacuation tube and the capsule was determined by microstructural analysis. The capsules were prepared by cutting, polishing, and etching with V2A pickle to enhance the grain boundary visibility. For microscopic investigations, a light microscope Zeiss Axio Imager, as well as a FEI Helios Nano Lab Dual Beam electron microscope, were used. The Image analysis was conducted with the software ImageJ [19]. Additionally to the microstructural analysis, the argon content within the capsules was measured with a Thermo Fisher Scientific Trace GC 600 quadrupole mass analyzer according to the Swedish SS 118000:2018 standard [20]. The sample mass for argon measurements was 0.5 g - 1.0 g and the lower detection limit was 0.049 ppm.

Results and discussion

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One out of the five TIG-welded HIP capsules could not be consolidated and the powder filling remained nearly unchanged in the course of the HIP process. Therefore, the microstructure and argon content could not be analyzed and determined. The four other joints were gas-tight and suitable for the PM-HIP process. The microstructure of one representative TIG-welded HIP capsule is depicted in Figure 3. The capsule deformed almost rotationally symmetric around the central axis with necking in the thinner area, which is intended to act as the brazing gap for subsequent brazing experiments. Unetched microsections, as shown in Figure 3 b), revealed little to none porosity within the compacted powder filling. However, a high degree of oxides was formed along the prior particle boundaries (PPB). The specimen in Figure 3 had an entrapped argon content of 0.63 ppm, and the overall argon content of the four TIG-welded capsules was 1.3 ppm. All results from the argon analysis are depicted in Figure 6 b). Etching of the AISI L6 bulk revealed a fine-grained and needle-shaped martensitic microstructure, as shown in Figure 3 c). The interface between the AISI A11 capsule and the L6 filling showed a good bonding with very few defects in the form of voids and local separation, as depicted in Figure 3 d).

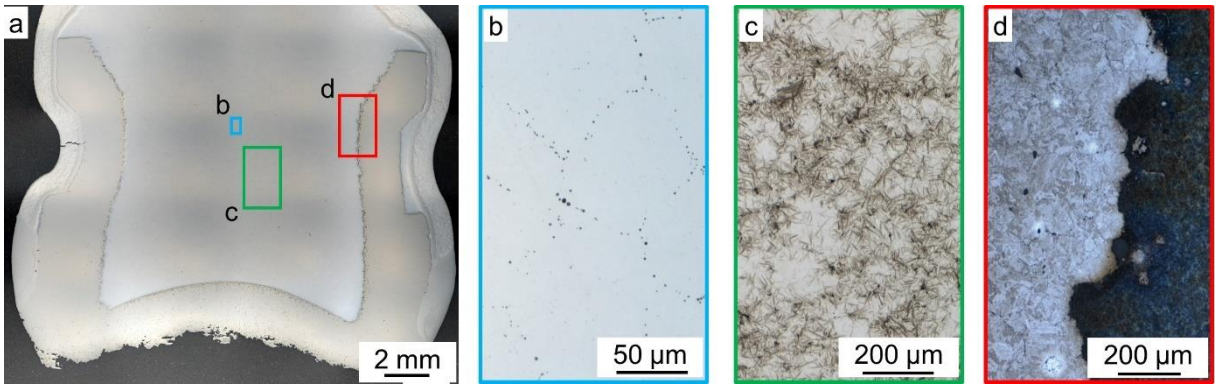


Figure 3. a) TIG-welded HIP capsule after consolidation, b) unetched L6 microstructure, c) etched microstructure, d) interface

Similar results were obtained when the evacuation tube was bonded with the TLP process and the commercially available filler material BNi-5. One HIP capsule could not be consolidated by the HIP process, indicating a non gas-tight bond. The remaining four specimens were consolidated successfully. Similar to the welded HIP capsules, a significant amount of oxides was formed along the PPB's, as indicated in Figure 4 b). The entrapped argon content within this specimen was 0.49 ppm, while the average argon content within the capsules, influenced by a single outlier, was 2.39 ppm. The bonding zone between the capsule and the evacuation tube is depicted in Figure 4 d). Due to the significant amount of silicon within the nickel-base BNi-5 filler material, intermetallic precipitates were formed. SEM investigations revealed two types of precipitates in the form of dark and light grey plate-like phases. The light grey precipitates could be identified by EDS analysis as a mixed nickel and silicon phase, while the dark grey precipitates were carbides enriched with chromium, nickel, silicon and vanadium.

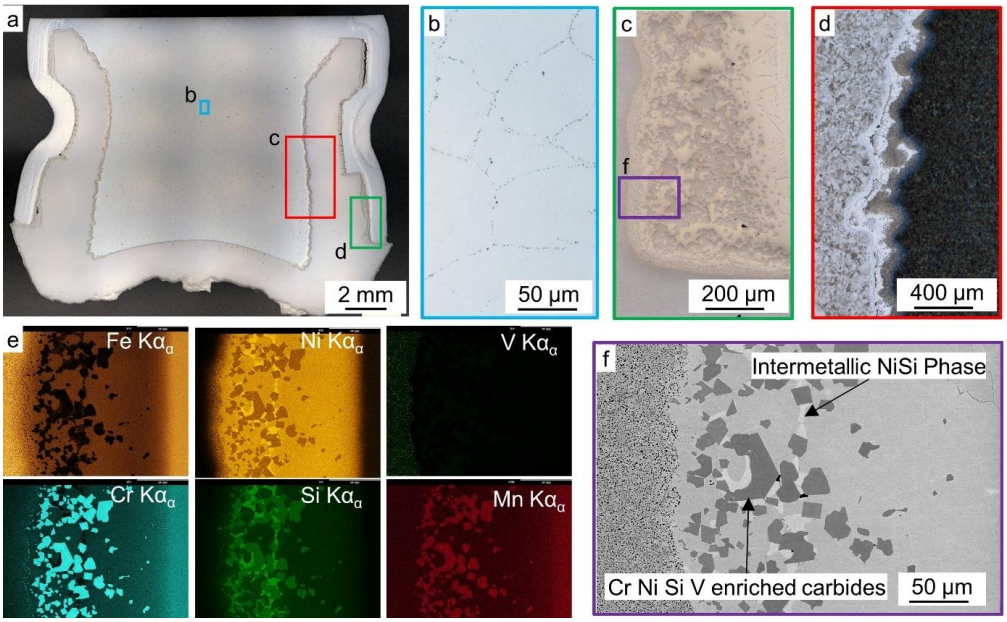


Figure 4. a) TLP (BNi-5) bonded HIP capsule, b) unetched L6 microstructure, c) etched fusion zone, d) interface capsule/filling, e) EDS mapping of fusion zone, f) SEM image of fusion zone

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Figure 4 d) shows the interface between AISI L6 and the AISI A11 shell. Due to the TLP process principle, where liquid phase is formed during the process, filler material entered into the A11 shell and acts as diffusion barrier between the powder filling and the capsule. Therefore, no bonding between the two materials is evident.

The results of the TLP bonding process with the AISI A11 + 3.5 wt.% silicon filler material are depicted in Figure 5. Three out of five joints were destroyed during the process development for the inductive heating due to overheating and subsequent melting. The remaining two capsules could be successfully filled with powder and consolidated by HIP. Again, oxides are formed along the PPB's within the AISI L6 bulk. The average entrapped argon content was 0.63 ppm.

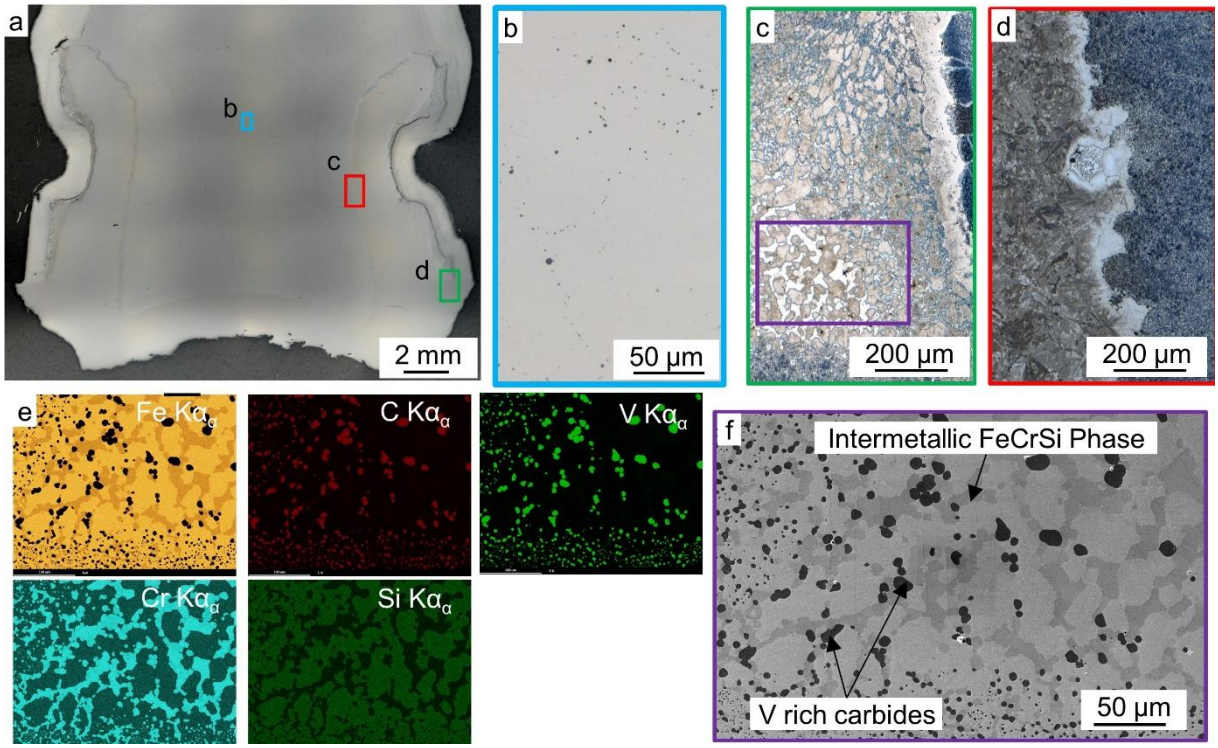


Figure 5. a) TLP (A11 + 3.5 wt% Si) bonded HIP capsule, b) unetched L6 microstructure, c) etched fusion zone, d) interface capsule/filling, e) EDS Mapping of Fusion zone, f) SEM image of fusion zone

The brazing zone and interface between the HIP capsule and the evacuation tube is depicted in Figure 5 c). Again, intermetallic precipitates in the form of dark and light grey phases were formed. Additionally, vanadium rich carbides were formed in the fusion zone, visible as black spots within the microstructure. The transition zone between the HIP capsule and the powder filling showed a strong diffusion zone, visible as the white area in the center of Figure 5 d).

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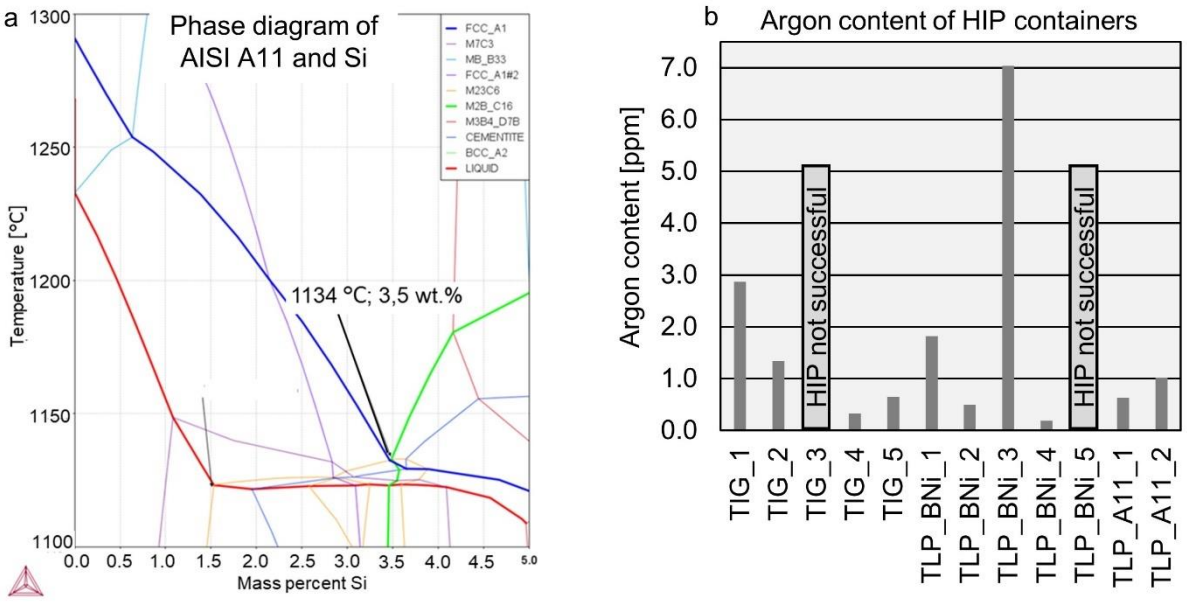


Figure 6. a) Phase diagram of AISI A11 and Si b) argon content of investigated HIP capsules

Conclusion

The feasibility of different techniques for joining of additively produced PM-HIP capsules with poor weldability and evacuation tubes made of austentic steel was proven within the scope of this research. Concerning the process reliability, only four out of five joints were gas-tight when TIG welding and furnace TLP bonding were used. The failure probability of up to 20 % can be attributed to the production conditions in laboratory scale. By improving the process, a much lower reject rate will be achievable.

All investigated joints showed a significant number of oxides along the prior grain boundaries. This might affect the resulting mechanical properties negatively, especially regarding the materials toughness and the resistance to crack propagation. Additionally, a high amount of entrapped argon within the capsules was determined, which in some cases exceeded 2 ppm and reached almost 7 ppm. These specimens do not comply with the requirements in SS 118000:2018. As argon contamination cannot be accepted in industrial applications, further investigation is needed in order to improve the process.

Nevertheless, TLP bonding with commercially available filler materials as well as custom filler materials seems to be a practicable alternative to welding, despite the formation of brittle intermetallic phases in the fusion zone. An unexploited potential lies in joining technology with the A11 + 3.5 wt% Si braze filler. Since the formed interface is rich in carbides, the capsule of complex and large components can consist of several additively and conventionally manufactured and joined sections and remain on the component as a functional, wear-resistant layer.

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