



Molybdenum Copper MMC for Additive Manufacturing of Thermal and Structural Components

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1 Introduction

The holistic approach of an extensively additive manufactured laser system requires the research into the generation of optical parts as well as structural and thermal components. This includes mountings and heat sinks for crystalline active laser mediums, for example Nd:YAG (neodymium-doped yttrium aluminum garnet). Conventionally, these heat sinks are based on form-fitting copper mountings for heat dissipation and indium foil to overcome roughness [1, 2] and expansion with limited functionality due to the production techniques.

Additive manufacturing of metallic materials, however, allows for a variety of innovative application possibilities due to further degrees of freedom. Regarding topology optimization and functional integration, structures can be generated where conventional production techniques obtain unsuitable or restricted manufacturability correlating with extensive effort.

The potential range of materials, though, is mainly limited to several powder materials offered by the manufacturers of these laser metal deposition systems. Therefore, the processed materials represent a compromise for the envisaged application often while powder materials have significant benefits due to the targeted adjustment of their composition in fact. Thus, specific characteristic profiles can be achieved.

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As a part of the joint project, wGROTESK (material development for additive manufacturing of optical, thermal and structural components) deals with the selection as well as the targeted adjustment of an application-specific material whilst ensuring and developing the processing by laser metal deposition (LMD). The additive manufacturing of mounting structures for YAG requires metallic materials combining suitable thermal conductivity, melting points and thermal expansion coefficients to avoid thermal damage resulting from thermal induced stress as well as a sufficient coupling of YAG and alloy to allow for a material closure.

The conventional mounting relies on copper since it offers a remarkable thermal conductivity. For the additive process, the melting point represents a major challenge due to the possibility of thermal damage to the YAG. Furthermore, the thermal expansion coefficient is unsuitable and the absorption for laser systems with infrared wavelengths, well established in small and medium-sized businesses, is insufficient.

The objective of the following work is the development and targeted adaption of a metallic alloy with a low melting point and a thermal expansion corresponding to that of yttrium aluminum garnet, while ensuring the bonding of the two incompatible materials through adequate chemical interaction.

In this context, the material development provides the approach to define a copper-based pseudo alloy for realization in an industrial laser process. The combination of copper and molybdenum allows for sufficient thermal expansion coefficients while a further adaption of the copper alloy presents the opportunity for an extensive adjustment of the material properties to meet the requirements of processing and implementation.

2 State of the Art and Scientific Bases

2.1 Challenge of Infrared Absorption of Copper

Laser sources with infrared wavelengths are largely disregarded for additive processing of copper. The main reasons for this are both the wavelength-dependent absorption and the thermal conductivity.

Pure copper shows an absorption of about 5% for wavelengths of around 1064 μm at room temperature (RT) (cf. Fig. 1 according to [3]). In combination with the high thermal conductivity, this means that only a small proportion of the laser power can be used for melting and the induced heat is immediately dissipated from the process zone. At the same time, the absorption abruptly increases directly before the melting point (MP) is reached, resulting in spatter formation and an unstable process.

Other wavelengths seem more suitable for processing copper in the laser-based additive process. Therefore, lasers with wavelengths in the green spectral range are often used to achieve improved process quality. However, these beam sources are considerably more

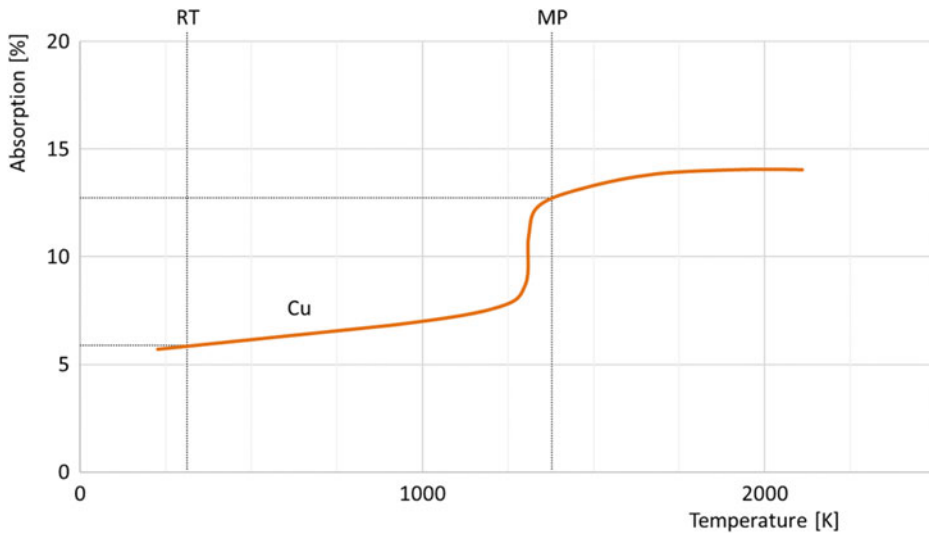


Fig. 1 Temperature dependence of the absorption for infrared wavelengths of pure copper according to [3]

expensive than IR laser sources. At the same time, IR lasers are more widespread in industry, especially among small and medium-sized companies.

Molybdenum copper pseudo alloys present a substantial benefit. The application of a powder mixture of molybdenum and copper offers the advantage of a significantly higher absorption for infrared wavelengths at room temperature (cf. Fig. 2 according to [3, 4]). This makes it possible to use reduced laser power for heat input and heat conduction to the copper in order to transfer it indirectly into the molten phase. This enables an increase in processability and thus an improved process quality.

2.2 Metal Matrix Composites and Pseudo Alloys

The term metal matrix composite generally refers to the combination of a metallic matrix with embedded ceramic particles or fibers. In addition, however, the combination of metallic matrix and high-melting metallic particles are also included.

The compound of matrix and particles primarily serves to achieve defined property profiles of the material used. This includes not only coefficients of thermal expansion for use in heat sinks, but also implementations in applications with high electrical conductivity [5, 6]. At the same time, there are also application possibilities where the ductility of metals is to be combined with the hardness of ceramics [7].

While metallic materials with low thermal expansion coefficients have been used for additive manufacturing for many years, they are primarily based on iron-nickel alloys

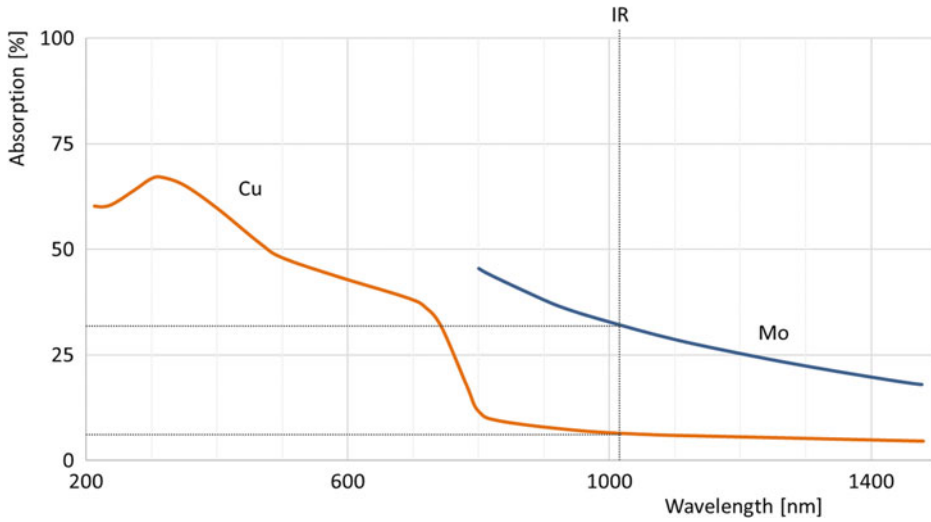


Fig. 2 Wavelength dependence of the absorption of pure copper and molybdenum according to [3, 4]

known as Invar or Kovar [8, 9]. The thermal expansion coefficient of these alloys at room temperature is between 1 and $5 \cdot 10^{-6} \text{ K}^{-1}$ depending on the composition [10]. However, at temperatures above $300 \text{ }^\circ\text{C}$, this increases significantly to over $12 \cdot 10^{-6} \text{ K}^{-1}$ [11]. At the same time, such alloys exhibit melting temperatures above $1400 \text{ }^\circ\text{C}$. Pseudo alloys offer the advantage of constant thermal expansion coefficients over a wide temperature range. In addition, only the matrix material has to be converted into the molten state.

In particular, materials combining copper and molybdenum as well as copper and tungsten also offer advantages in terms of thermal conductivity [12–14].

The production of pseudo alloys can be accomplished in different ways like layered composite materials [13], sputtered copper film with minor proportions of molybdenum [15], spark plasma sintering [16] or high pressure torsion [17]. Other approaches are based on powder metallurgy and sintering [5, 14, 18, 19]. Another conventional process route involves a two-step process in which the molybdenum grains are first sintered and subsequently the porous body is infiltrated with copper [20, 21].

The application of metal matrix composites and pseudo alloys by laser metal deposition process is a subject of research. Titanium alloys with minor proportions of Al_2O_3 nanoparticles and AlSi10Mg alloy with up to 2 vol% of tungsten carbide have been processed [22, 23].

However, additive processing of MMCs and pseudo alloys is commonly limited to material combinations with high percentage of matrix material and less bounded particles.

3 Experimental Approach

3.1 Specimen Generation at Quasi-static Ideal Thermal Conditions

In addition to the experiments for process development of the additive manufacturing process, which are carried out on a laser powder deposition system with a 680 W diode laser, the material development and adaptation is primarily performed at quasi-static thermal conditions in a furnace process. Due to the different process conditions, the influence of the divergent heating and cooling conditions can be compared and qualified. In this way, time- and temperature-dependent procedures such as diffusion processes can be represented in the different manufacturing methods.

The furnace-based generation are performed with a DataPhysics OCA drop shape analysis with a mounted furnace of the type HTFC 1800. Temperatures of up to 1800 °C can be reached under inert atmosphere with contemporaneous camera-based recording of the process zone. This enables simultaneous observation of the melt forming during the heating of the metallic powders. The experiments are carried out in an argon atmosphere at a flow rate of 200–250 l/h. The temperature is set individually above the melting temperature of the alloy applied.

This allows not only the generation of samples for microstructure analysis as well as the determination of thermal and mechanical properties but also for characterizations of the coupling of alloy and YAG. By detecting the contour of the formed melt drop, the contact angle can be automatically calculated by the definition of the substrate surface. The contact angle is directly associated with the coupling and wetting behavior. Small angles represent a proficient wetting of liquid and substrate. Thus, the objective of suitable bonding can be verified quantitatively.

Moreover, the melting temperature of the alloy can be determined by thermocouples.

Micro sections of the generated specimens are subsequently examined by means of reflected light microscopy and field emission scanning electron microscopy with regard to the formed microstructures and boundary layers.

3.2 Material Adaption

In general, the material adaptation for laser metal deposition can be differentiated according to the selected feed of the filler material. For wire-based materials, a course adjustment of the properties can be achieved by an adequate selection of the material of the wire. The usage of coatings applied with physical vapor deposition allows for a more precise adaption. Thereby, a combination of several layers is conceivable. During the manufacturing process, in situ alloying of all wire and coating components takes place. However, the application of pseudo alloys requires a complex production of cored filler wires.

In this respect, powder-based processing in the LMD process represents an improved possibility for short-term adjustments. The essential adaptation is made by the suitable choice of the composition of the material. Apart from that, the admixture of additives and reactive grinding are possible as adaptations. Due to the specific adjustability of the composition, this processing offers significant advantages for achieving the defined property profiles.

Accordingly, the focus of material development is on powder-based processing with rapidly adaptable compositions. This short control loop allows a fast response to the measured properties of the material. This makes it possible to meet the objective of a targeted adaptation of the material's properties in order to realize the desired approximation to the additively manufactured laser system. The appropriate choice of the added elements can ensure the development of the alloy in terms of low melting point, suitable thermal expansion coefficients and coupling of the incompatible material groups through a sufficiently large chemical interaction.

4 Challenges in the Development of a Suitable Pseudo Alloy

4.1 Incompatibility of Material Groups

The metallic molybdenum copper pseudo alloy and the crystalline YAG represent fundamentally different material groups. With regard to the thermal expansion of the materials, the pseudo alloy is intended to overcome this discrepancy. Therefore, the near linear course of the thermal expansion coefficient of YAG must be met as well as possible.

Another challenge, however, is the coupling of YAG and metal. The material groups have a completely different structure. The objective of additive bonding of the crystalline YAG is not a form-fit connection, but explicitly a material-fit connection of the two materials. Consequently, it is inevitable that a suitable coupling of the molten metal to the YAG takes place, which also remains after cooling and solidification of the alloy. At the same time, a similar thermal expansion is required, since thermally induced stresses should be avoided as far as possible so that the risk of thermal damage is prevented.

4.2 Materials Morphology

The conventional processing of molybdenum-copper composites to date is based on the sintering of molybdenum particles with the subsequent infiltration of the sintered body with the molten copper. The resulting microstructure is formed as shown in Fig. 3a. In principle, this process can also be transferred analogously to a copper matrix.

The challenge in additive manufacturing, taking into account the thermal boundary conditions, lies in transferring the two-stage process route into a single-stage process. At

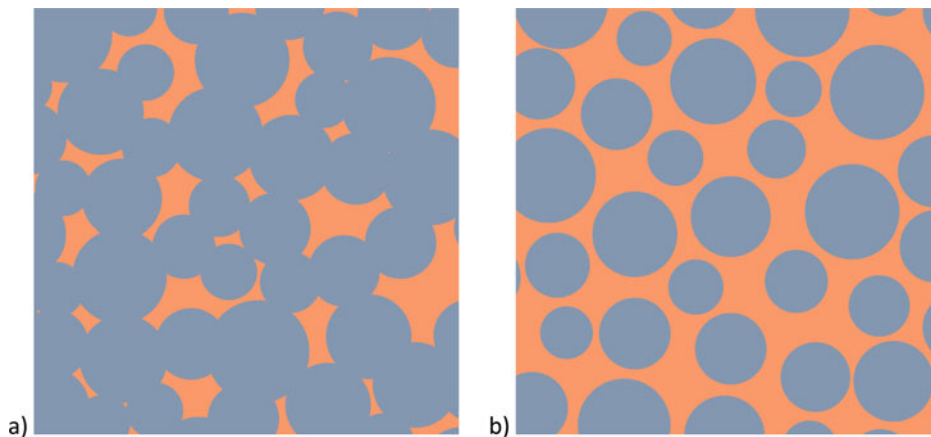


Fig. 3 Schematic microstructure of the molybdenum copper pseudo alloy **a** sintered molybdenum particles after conventional processing **b** molybdenum particles integrated in copper matrix

around 1000 °C, the process temperatures are far below the temperatures required for sintering the molybdenum particles. Consequently, only the matrix material is transferred to the molten phase in which the molybdenum particles are bonded. The formation of the microstructure is similar to that in Fig. 3b).

For the targeted adaption of the material, the specimen generation is initially focused on processing by optical contact angle goniometers. The installed furnace chamber allows for inert atmosphere combined with quasi-static thermal conditions. Figure 4 shows a molybdenum copper pseudo-alloy with a molybdenum content of about 70 wt%.

The high number of imperfections (black areas) is striking, indicating that the wetting of the copper on the molybdenum and capillary effects between the molybdenum particles are insufficient. It is therefore essential for suitable processing that the wetting is increased substantially.

5 Results

5.1 Wetting of Matrix Material on Molybdenum

Pure copper as a matrix material reveals significant imperfections during processing with regard to the microstructure. To increase mold filling, the use of copper phosphorus alloys is being investigated. Alloys of this type with about 8 wt% phosphorus are used as solders due to their capillary-active, good flow properties [24, 25]. Figure 5 shows the significant improvement of mold filling when CuP8 is applied to the molybdenum.

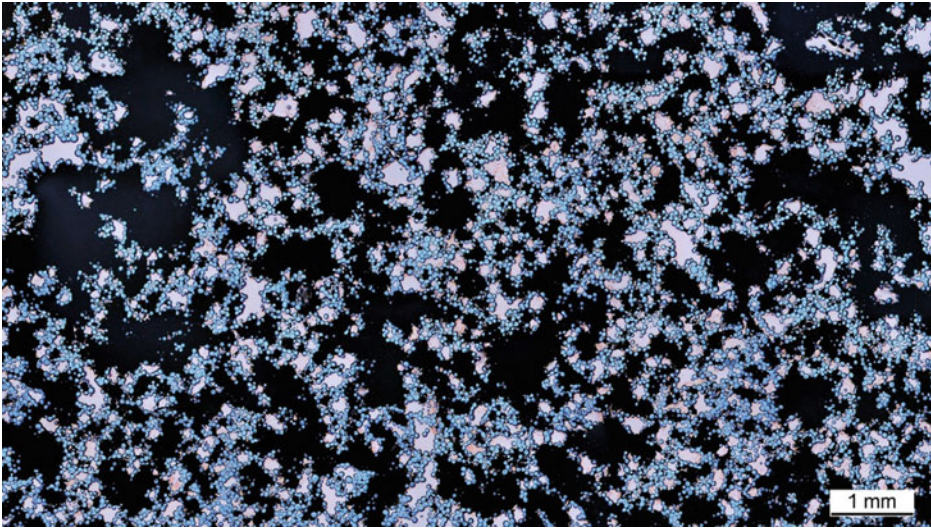


Fig. 4 Microstructure of furnace-generated pseudo alloy with 70 wt% molybdenum and copper balance

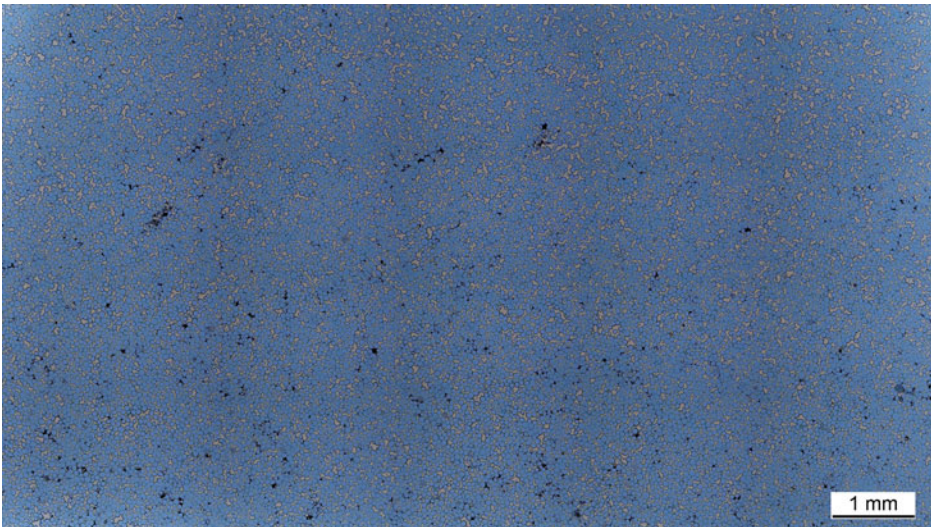


Fig. 5 Microstructure of furnace-generated pseudo alloy with 70 wt% molybdenum and CuP8 balance

The quantification of the impact of phosphorus can also be demonstrated by means of contact angle measurements. Figure 6 shows the wetting of pure copper on molybdenum with a contact angle of about 40°. Increasing the phosphorus content in the copper alloy leads to successively improved wetting on the molybdenum substrate. Figure 7 shows that already 1 wt% phosphorus in the copper leads to a significant difference while 8 wt% phosphorus results in ideal wetting.

In addition to improved wetting, the copper-phosphorus system offers the advantage of a lower melting point. At the eutectic point, this is around 714 °C and thus considerably lower than that of copper (1084 °C) [26]. Consequently, the risk of thermal damage during additive imprinting of the YAG is significantly reduced as a result of lower thermal stresses.

This provides the basis for the application of the pseudo alloy with regard to the sufficient chemical wettability of the refractory metal molybdenum and the low melting point of the matrix material.



Fig. 6 Microstructure of wetting of pure copper on molybdenum substrate

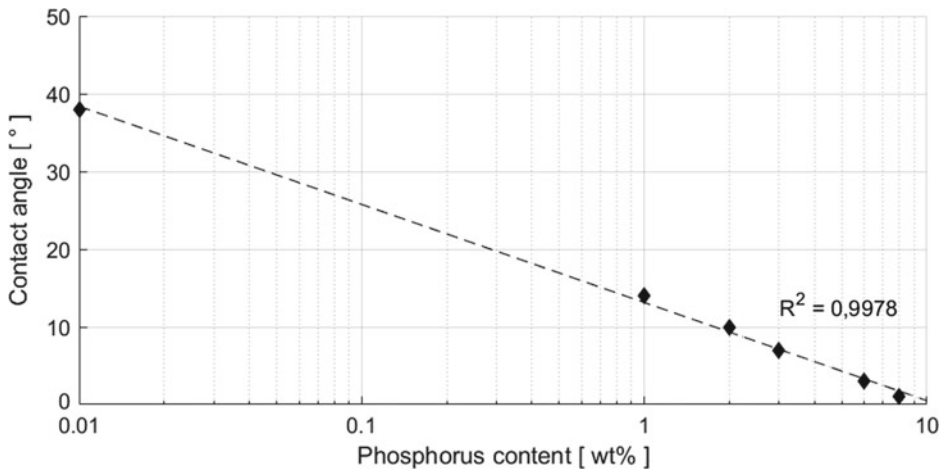


Fig. 7 Contact angle of copper alloy on molybdenum substrate depending on phosphorus content

5.2 Coupling of YAG and Alloy

While the wetting behavior of a metal–metal combination requires only limited adaptations, the combination of metal and nonmetal causes a major challenge. The realization of a sufficient coupling of these theoretically incompatible materials is implemented due to the application of suitable additives solely.

The characterization of the wettability is examined by contact angle measurements. Smaller contact angles correlate with improved wetting along with better bonding.

For the measurement of the contact angle of alloy on YAG, only the matrix material is applied. On the one hand, this is due to the high molybdenum content, which otherwise makes the measurements considerably more difficult; on the other hand, only the matrix material becomes molten and allows interaction with the crystalline YAG.

As expected, the contact angle is very high with the combination of copper and YAG as a result of which no sufficient bonding takes place. Values of about 110° are measured both with pure copper and when using the copper-phosphorus alloy. Furthermore, there is no sufficient bonding since the melt drop can be easily detached when cooled.

In previous experiments, it was demonstrated that a titanium coating of the YAG by a preceding physical vapor deposition process can lead to a significant improvement of the wetting [11].

Regarding improved processing properties, the objective is to avoid the indirect route via the coating process, reaching an increased wetting by means of a direct admixture of additives. The addition of titanium allows significantly lower contact angles, but results in pronounced oxidation on the surface of the melt droplet despite an argon atmosphere.

The wetting can also be increased by adding manganese to the copper-phosphorus matrix. Thus, the contact angle is about $80\text{--}90^\circ$ (see Fig. 8a)). Although manganese, like titanium, has a high affinity for oxygen, oxidation does not occur. Since oxygen is a component of the YAG, this leads to the formation of a metallization and the associated improvement in wetting [cf. 24].

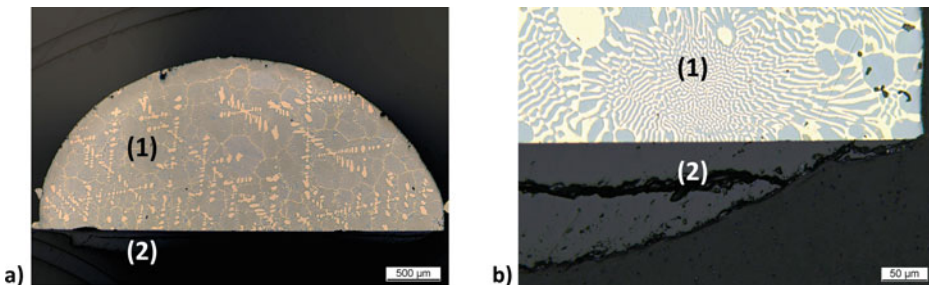


Fig. 8 Microstructure of wetting: CuPMn alloy on YAG **a** and enlarged display of detachment within YAG; explanatory note: (1) CuP alloy, (2) YAG

During cooling, the solidified metal droplet is detached from the YAG substrate. However, the microsection reveals a detachment within the YAG along with remaining adhesions of the alloy, indicating a sufficient bonding (see Fig. 8b)). Due to the unadapted thermal expansion coefficients issued from absent molybdenum admixture, high mechanical stresses are induced in the YAG and damage results.

By adding manganese, the objective of a suitable bonding of non-metallic and metallic material can be achieved much better compared to the use of titanium as an alloying element.

5.3 Thermal Expansion Coefficients

One of the main reasons for selecting the molybdenum copper pseudo alloy is the low thermal expansion coefficients that can be achieved with the conventionally processed composite. However, the innovative approach relinquishes the advantages of sintered connections to ensure low melting points. Nevertheless, the low thermal expansion coefficients of the YAG must be met.

Figure 9 shows the measured thermal expansion coefficient of YAG, copper and molybdenum as well as several compositions of the pseudo alloy generated via furnace process. The values of the pseudo alloy are marginally higher than those of the YAG. However, the curve shows a nearly parallel course. Consequently, there is a slightly higher shrinkage in

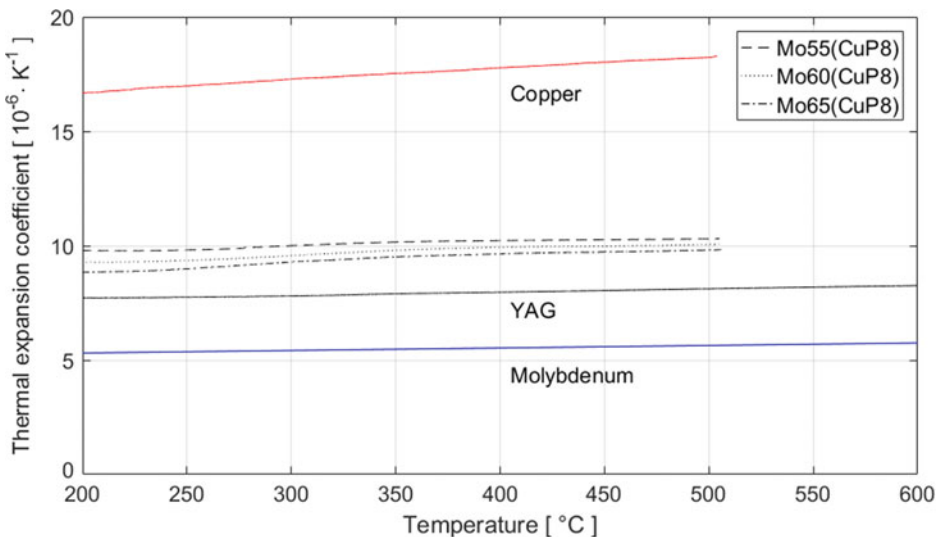


Fig. 9 Thermal expansion coefficients of pure materials and selected pseudo alloys with different contents of molybdenum

the alloy than in the crystal when cooling during the manufacturing process, with the difference showing a linear progression. As a result, low compressive stresses are expected within the YAG.

Furthermore, the graph demonstrates the relation between thermal expansion and molybdenum amount, while a higher percentage of molybdenum leads to a reduced thermal expansion coefficient.

Compared to the literature values for copper-molybdenum composites with sintered connections between the molybdenum grains, the values determined for the process route shown here are slightly higher. However, a targeted adaptation of the thermal expansion can be demonstrated via the adjustment of the composition.

5.4 Thermal Conductivity

Various aspects must be taken into account when determining the thermal conductivity of such pseudo alloys. Due to the high molybdenum content, measurement values of pure copper cannot be expected. The application of CuP8 instead of copper entails a further limitation.

Figure 10 shows the thermal conductivity of pure copper, CuP8 and MoCuP compounds with 40–60 wt% molybdenum, respectively, logarithmically plotted against the temperature. The thermal conductivity of molybdenum amounts to $140 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$.

The difference between pure copper and CuP8 is significant. While the specimen of pure copper achieves measurement values of $250\text{--}290 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, the copper phosphorus alloy achieves not more than a tenth of the thermal conductivity. The measured values of the pseudo alloy amount to $35\text{--}50 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ depending on the content of molybdenum.

5.5 Transfer of Processing to Additive Manufacturing

Essentially, it was demonstrated that the adaptation of a molybdenum copper pseudo alloy can be successfully implemented with regard to the intended application in furnace-based sample generation. For the transfer to the additive laser metal deposition process, the absorption for infrared wavelengths is of interest to ensure process-reliable manufacturing.

Figure 11 shows measurements of various metal powders for wavelengths of 940–980 nm, which allow a qualitative classification of the absorption with respect to copper powder. Molybdenum shows more than twice the measured values than copper does, while the absorption of the copper phosphorus alloy is greater by a factor of three at least. The pseudo alloy falls in between with regard to the molybdenum content.

Consequently, the pseudo alloy allows significantly improved absorption for infrared wavelengths and is more suitable for additive processing, inevitably requiring process

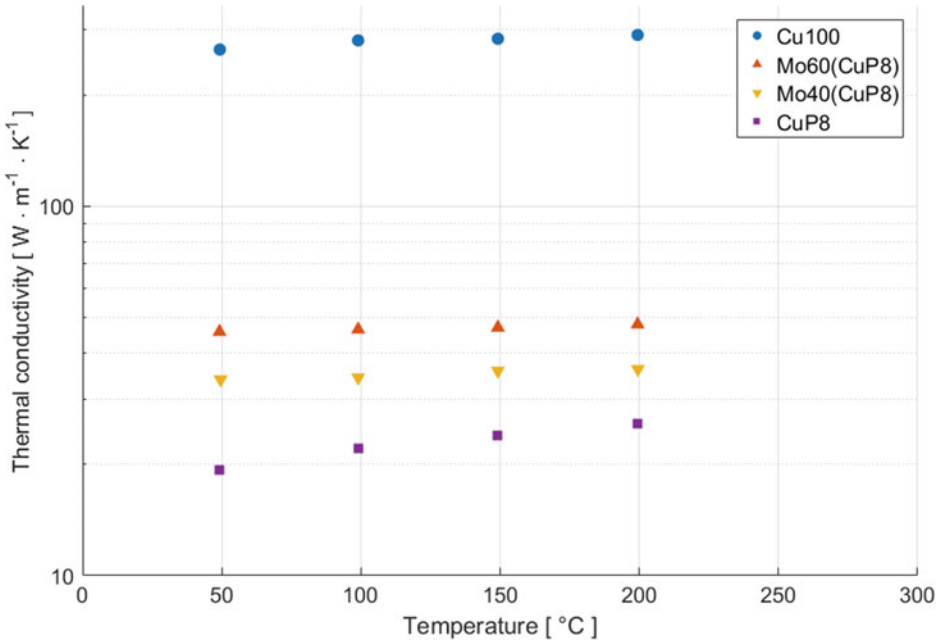


Fig. 10 Thermal conductivity of pure copper, CuP8 and MoCuP compounds as a function of temperature

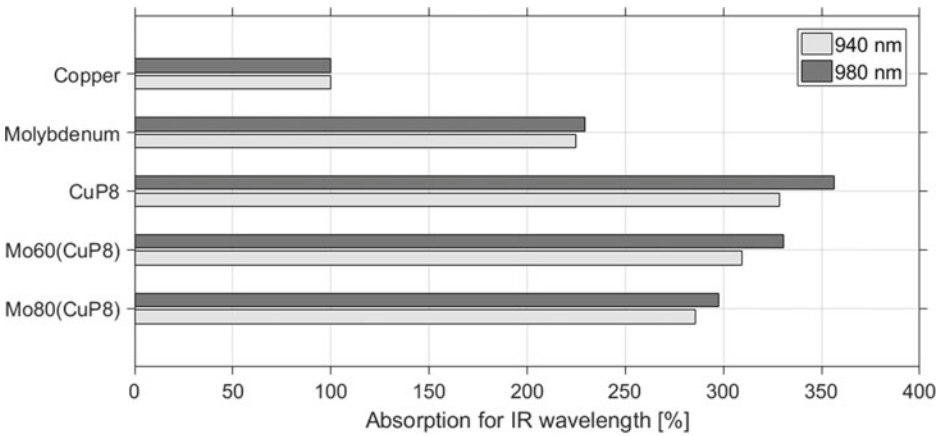


Fig. 11 Qualitative comparison of the absorption for infrared wavelengths for selected powder materials

development. During processing, it must be ensured that the temperatures are within the required range to achieve both melting of the matrix material and to prevent damage to the YAG due to excessively high temperatures. At the same time, the molybdenum particles should be distributed uniformly in the matrix in combination with a small quantity of imperfections in the structure.

In the following chapter, the development of the process strategy for laser-based additive manufacturing of this alloy will be discussed in more detail. At this point, the focus is exclusively on the fact that the pseudo-alloy can be successfully processed additively and multilayered structures with a thickness of several millimeters can be generated. Figure 12 shows the resulting microstructure with molybdenum particles bound in a copper phosphorus matrix.

While furnace-based processing has advantages with regard to the risk of damage to the YAG due to the thermal conditions, additive processing requires significant adjustments. In principle, it can be demonstrated that the processing of the pseudo alloy by laser metal deposition can be realized in a process-safe manner. For a multi material connection, on the other hand, precautions must be taken to avoid thermal damage. This requires that both the YAG is suitably prepared and the process is suitably adapted. Due to titanium coating on the YAG applied by physical vapor deposition, bonding to the pseudo alloy can be achieved much more easily while processing. By preheating the process zone, high temperature gradients and the risk of thermal shock can be eliminated. The use of

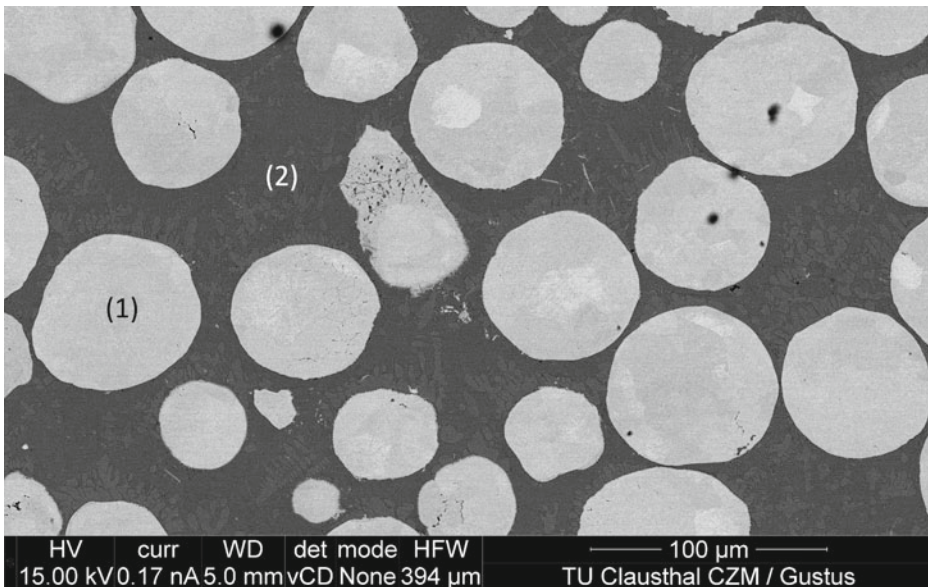


Fig. 12 FESEM picture of molybdenum copper pseudo alloy generated by additive manufacturing; explanatory note: (1) molybdenum, (2) CuP matrix

stepped seams enables uniform heat input distributed over the entire circumference. Thus a suitable additive binding and a sufficient process quality can be obtained. Initial tests on the imprinting of crystalline materials, taking into account the precautions described, achieved satisfactory results.

5.6 Formation of Microstructure

The different process conditions of furnace-based manufacturing and additive manufacturing inevitably lead to differences in microstructure formation. The quasi-static thermal conditions in the furnace allow for more extensive chemical interactions and diffusion processes in contrast to the additive process where cooling is much faster due to the high temperature gradients. This is evident by examining the microstructures of both types of specimen. The copper phosphorus matrix exhibits a structure consisting of Cu_3P and (Cu) phases.

In the furnace process, a very pronounced phosphorus-rich phase seam is formed around the molybdenum particles. At the same time, Cu_3P bypasses form between the particles (Fig. 13a)). The rest of the matrix consists largely of the low-phosphorus (Cu) phase. The thermal conditions allow for long diffusion paths.

Due to the high temperature gradients of the additive process, the lamellae consisting of Cu_3P and (Cu) form much finer (Fig. 13b)). These lamellae are also distributed throughout the matrix. At the same time, the phosphorus-rich phase seams around the molybdenum particles are much narrower.

A more detailed investigation of the interfaces of molybdenum particles and copper matrix by means of field emission scanning electron microscopy (FESEM) reveals an interaction of molybdenum and phosphorus. In the furnace-generated sample, a seam of molybdenum phosphorus phases of different composition (MoP , Mo_3P and Mo_4P_3) is detected (Fig. 14a)). In addition to the physical wetting, there is therefore also a chemical

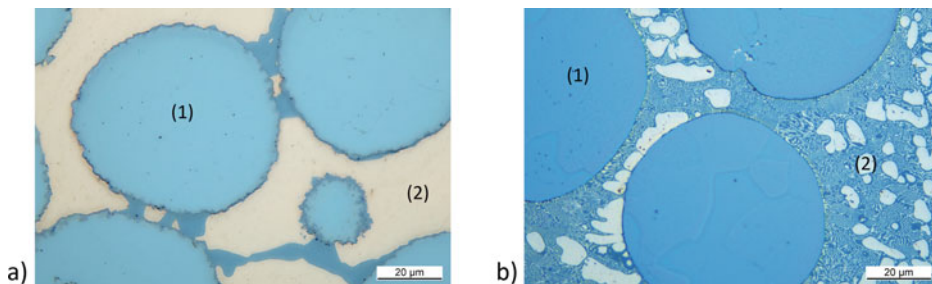


Fig. 13 Microstructure of pseudo alloy generated by furnace process **a** and additive manufacturing **b**; explanatory note: (1) molybdenum, (2) CuP matrix

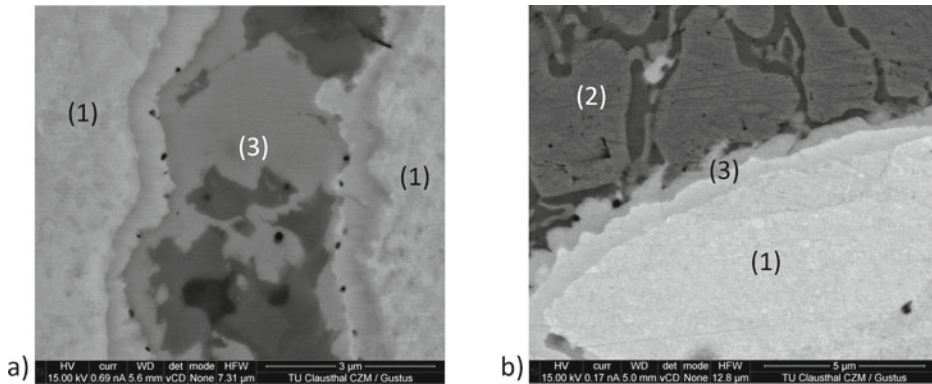


Fig. 14 FESEM picture of pseudo alloy generated by furnace process **a** and additive manufacturing **b**; explanatory note: (1) molybdenum, (2) Cu₃P matrix, (3) Mo_xP_y phases

interaction. Moreover, MoP bridges form between the molybdenum particles when these particles are close to each other.

Even though the high temperature gradients in additive processing significantly limit chemical interactions and diffusion processes, molybdenum phosphorus phases can also be detected. However, Fig. 14b clearly reveals that these phase seams are much smaller.

The presence of these seams therefore indicates chemical interaction between the components of the pseudo alloy. Consequently, there is inevitably a bonding of molybdenum particles and copper matrix.

6 Conclusion and Perspective

It can be shown that the transfer of molybdenum-copper composites to additive manufacturing is feasible, if the limitations of the additive process are considered. The pseudo alloy enables the properties to be adjusted according to the required profile. The combination of copper and molybdenum opens up wide-ranging possibilities for adjusting thermal expansion. The thermal expansion coefficients of the pseudo alloy show an almost parallel course compared to the YAG and can be finely adjusted by the molybdenum content.

The lack of sintered bridges is reflected in slightly higher expansion coefficients compared to conventional processing of the composites. At the same time, however, the innovative processing approach allows for the possibility of more complex structures and functional integrations that can only be implemented using additive manufacturing.

Depending on the selected process route, the microstructure differs significantly. Segregation within the copper phosphorus matrix is inevitable, but a larger seam of the phosphorus-rich phase forms around the molybdenum particles considering the furnace process. At the same time, the formation of Cu₃P bypasses between the particles occurs.

The rest of the matrix consists of low-phosphorus copper phase. Even though there are no sintered bridges between the molybdenum particles, it can be demonstrated that an interaction with the phosphorus from the matrix material takes place, since Mo_xP_y phase bridges between the particles can also be detected. In the additive process, the segregation of (Cu) and Cu_3P is finely lamellar throughout the matrix, with much less pronounced phosphor-rich phase seams around the molybdenum particles. The background to this is the much higher cooling rates and the associated shorter diffusion paths. The bond between molybdenum and matrix material is obviously based not only on physical wetting, but also on chemical interaction.

The phosphorus, which plays a significant role in bonding, also leads to a lower melting point compared to the use of pure copper as a matrix material. This results in improved suitability for the intended application of additive mounting of the YAG. The low melting point leads to lower thermal stress and consequently a reduced risk of thermal damage to the YAG.

On the other hand, it can be seen from the thermal conductivity of the CuP alloy compared with pure copper that the CuP alloy as the matrix material of the pseudo alloy exerts a significant influence on the low thermal conductivity. Nevertheless, the pseudo alloy exhibits sufficiently high thermal conductivity for application in the additively manufactured laser system.

The bonding of matrix and YAG can be specifically improved by adding titanium or manganese. With the former, however, the oxygen affinity is particularly high, resulting in oxidation at the surface of the solidified alloy.

Since it has been shown that the additive processing of the pseudo alloy is possible, the adaptation of the material allows a use for a multi-material connection to the YAG, and first tests for additive mounting of the YAG show successful results, the upcoming final step is the process-safe generation of a laser system with mounted YAG. At the same time, a final characterization of the pseudo alloy and the multi material compound is carried out.

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