ECCM 20

26-30 JUNE
2022
LAUSANNE
SWITZERLAND

Proceedings of the 20th European Conference on Composite Materials

COMPOSITES MEET SUSTAINABILITY

Vol 3 – Characterization

Editors : Anastasios P. Vassilopoulos, Véronique Michaud

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ESCM EUROPEAN SOCIETY FOR COMPOSITE MATERIALS

Proceedings of the 20th European Conference on Composite Materials ECCM20

26-30 June 2022, EPFL Lausanne Switzerland

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Oragnized by:

Composite Construction Laboratory (CCLab)
Laboratory for Processing of Advanced Composites (LPAC)
Ecole Polytechnique Fédérale de Lausanne (EPFL)

ISBN: 978-2-9701614-0-0
DOI: http://dx.doi.org/10.5075/epfl-298799_978-2-9701614-0-0

Published by :

Composite Construction Laboratory (CCLab)
Ecole Polytechnique Fédérale de Lausanne (EPFL)
BP 2225 (Bâtiment BP), Station 16
1015, Lausanne, Switzerland

https://cclab.epfl.ch

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https://lpac.epfl.ch

Cover:

Swiss Tech Convention Center
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Cover Design:

Composite Construction Laboratory (CCLab)
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And all those who helped, colleagues who reviewed abstracts and chaired sessions, and CCLab and LPAC students and collaborators who worked hard to make this conference a success.

Contents

INVESTIGATION AND CHARACTERIZATION OF THERMAL PROPERTIES OF A METAL MATRIX COMPOSITE REINFORCED WITH A METALLIC GLASS FOAM (NI60NB20TA20)

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Abstract: *The metallic glass presented in this work with alloy composition $Ni_{60}Nb_{20}Ta_{20}$ (at.-%) obtained its amorphous structure through a powder atomization process and was processed into an open-porous foam using laser powder bed fusion. Subsequently, the open-porous structure was infiltrated with an AlSi12 eutectic aluminum alloy by gas pressure infiltration. For manufacturing processes and applications, knowledge of thermal properties of the metallic glass foam as well as metal matrix composite is essential. Therefore, characteristics of thermal expansion were determined experimentally by dilatometry, specific heat capacity by differential scanning calorimetry and thermal conductivity by laser flash analysis. Thermal expansion as well as specific heat capacity are measured for the metallic glass foam as well as infiltrated composite. Laser flash analysis method was applied to the infiltrated composite only, as this method is not suitable for an open-porous structure. Thermal investigations revealed a relaxation in the metallic glass, which was investigated in detail.*

Keywords: metallic glass; metal matrix composite; thermal expansion; specific heat capacity; thermal conductivity

1. Introduction

Compared to crystalline metals, metallic glasses exhibit remarkable properties such as high strength, hardness, elastic strain limit due to their amorphous structure [1–3]. Conversely, they also exhibit low toughness and high susceptibility to brittle fracture, making them less qualified for the use as monolithic structural components [4]. To compensate for brittleness, metallic glasses are increasingly used as a reinforcing phase in a hybrid material, such as metal matrix composites (MMC) with interpenetrating structures. This requires that the metallic glass has an open-porous foam structure so that it can be infiltrated with a metal that has more ductile properties [5, 6]. The metallic glass used in this work with alloy composition $Ni_{60}Nb_{20}Ta_{20}$ (at.-%) obtained its amorphous structure through a powder atomization process and was processed into an open-porous foam using laser powder bed fusion (LPBF). Subsequently, the open-porous structure was infiltrated with an AlSi12 eutectic aluminum alloy by gas pressure infiltration. Due to the high crystallization temperature of the metallic glass $Ni_{60}Nb_{20}Ta_{20}$ alloy (721 °C [7]) and the low melting temperature of the AlSi12 alloy (577 °C [8]), it was possible to select a corresponding process temperature of 660 °C to maintain the amorphous structure of the metallic glass. For manufacturing processes and applications, the knowledge of thermal properties of the metallic glass foam as well as the MMC is essential. Therefore, the characteristics of thermal expansion were determined experimentally and characterized by dilatometer measurements, specific heat capacity by differential scanning calorimetry (DSC) and

doi:10.5075/epfl-298799_978-2-9701614-0-0

thermal conductivity by laser flash analysis (LFA). The thermal expansion as well as specific heat capacity are measured and investigated on the metallic glass foam as well as on the infiltrated composite. The LFA method was applied to the infiltrated composite only, as this method is not suitable for an open-porous structure. In addition, the thermal investigations revealed a relaxation in the metallic glass, which was also investigated in detail. Metallic glasses are formed by supercooling a liquid melt at very high cooling rates (10^2-10^8 K/s) [9, 10]. During this process, free volume is frozen, and the metallic glass is in a thermodynamic metastable state. Upon reheating (below crystallization temperature), a relaxation process begins. Thermally activated diffusion closes the free volume and a thermodynamic equilibrium is reached [11–13].

2. Material and experimental methods

2.1 Material

The metallic glass presented in this work with an alloy composition $Ni_{60}Nb_{20}Ta_{20}$ (TaNi39.1Nb20.7 wt.-%) obtained its amorphous structure through a gas atomization process by the company Nanoval GmbH & Co.KG (Berlin, Germany). The Nanoval process is crucible-free [14] and was carried out in an inert argon atmosphere. Rapid cooling causes the material to solidify into an amorphous powder with particle size $d_{50} = 44\,\mu m$. The powder was further processed into an open-porous foam with a measured reinforcement volume fraction of 37.7 % using LPBF. This Process was conducted by the research group “Production and Component Behavior” at the Institute for Applied Materials – Materials Science and Engineering, Karlsruhe Institute of Technology. Subsequently, the open-porous structure was infiltrated with an AlSi12 eutectic aluminum alloy by gas pressure infiltration. Due to the high crystallization temperature of the metallic glass $Ni_{60}Nb_{20}Ta_{20}$ alloy (721 °C [7]) and the low melting temperature of the AlSi12 alloy (577 °C [8]), a corresponding process temperature of 660 °C was selected to maintain the amorphous structure of the metallic glass. This results in an interpenetrating MMC with metallic glass as reinforcement phase.

2.2 Experimental methods

Investigations on thermal expansion of the $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam, the infiltrated MMC as well as the AlSi12 for comparison were carried out in a dilatometer type DIL 402 Expedis from Netzsch (Selb, Germany). The examined samples were cuboid-shaped with dimensions of 5 x 5 x 10 mm³ and plane-parallel surfaces. Due to the different structure of the $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam along and across the building direction caused by the LPBF process, the material is investigated in both directions regarding thermal expansion. Figure 1 shows on the

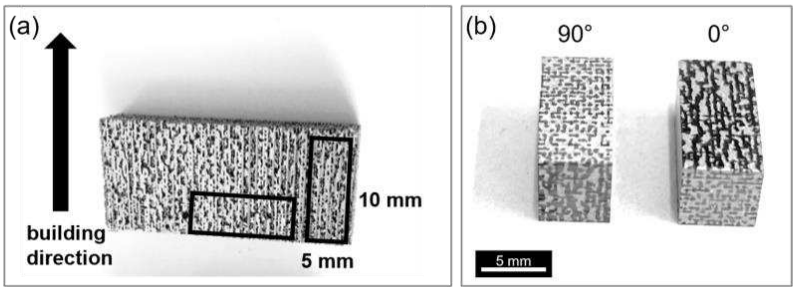

Figure 1. Samples for dilatometer measurements parallel (0°) and perpendicular (90°) to building direction. (a) Samples of $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam. (b) Samples of infiltrated MMC with AlSi12.

doi:10.5075/epfl-298799_978-2-9701614-0-0

left side the $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam and on the right side the MMC infiltrated with AlSi12.Measurements parallel to building direction are identified with 0° and perpendicular to building direction with 90°. Three thermal cycles of 20 °C to 500 °C with a constant heating and cooling rate of 5.5 K/min were carried out for each sample. The upper temperature limit was chosen to remain below crystallization temperature of the metallic glass and melting temperature of the AlSi12 (577 °C [8]). The contact force was set to 0.2 mN. To avoid oxidation all measurements were performed in an inert argon atmosphere. Therefore, the dilatometer was evacuated to a vacuum of 10^{-4} mbar and purged three times with argon to minimize the residual oxygen content. A reference measurement was performed with a Al_2O_3 sample to eliminate any effects of the testing device. All measurements were carried out and evaluated according to DIN 51045-1. The coefficients of thermal expansion (CTE) were evaluated in a range of 60 °C to 480 °C.

The specific heat capacity was determined by means of dynamic differential calorimetry in a DSC 214 Polyma from Netzsch (Selb, Germany) according to DIN 51007. For comparison and validation of the results, an Al_2O_3 sample was again used as reference. Accordingly, a sample size of approximately 1 x 2 x 3 mm³ of the $Ni_{60}Nb_{20}Ta_{20}$ foam and the MMC was chosen. The temperature program for the heat capacity measurements is composed of an isothermal start phase at 0 °C, a dynamic phase with constant heating rate of 20 K/min up to 200 °C and a final isothermal end phase at 200 °C. Since relaxation in metallic glasses has an influence on the specific heat capacity, the relaxation temperature T_r was first determined. For this purpose, four DSC measurements from ambient temperature to 500 °C (20 K/min) were carried out on one sample. All measurements were performed in an inert argon atmosphere.

Thermal conductivity was determined using laser flash analysis. The measurements were carried out with a LFA 1000 of the company Linseis (Selb, Germany) according to ASTM E 1461. In this method, the thermal diffusivity (a) of the material is measured, and the thermal conductivity (λ) is calculated with the density (ρ) and specific heat capacity (c_p) using equation 1.

$$\lambda = a\rho c_p \tag{1}$$

The specimens must have plane-parallel top and bottom surfaces and a defined thickness. This resulted in a specimen size of 10 x 10 x 1.6 mm³ as shown in Figure 2.

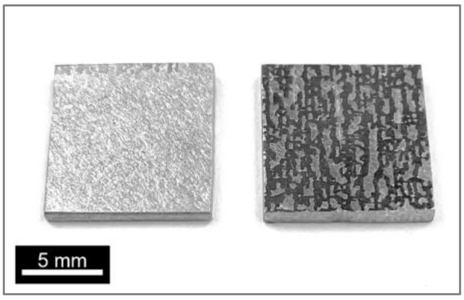

Figure 2. Samples for LFA. Left: Sample of AlSi12. Right: Sample of the infiltrated MMC.

Furthermore, the measurements require a continuous sample body, which is why the $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam could not be investigated with this method. Therefore, the measurement was additionally performed on an AlSi12 sample for comparison.

3. Results

3.1 Thermal expansion

Figure 3 shows the temperature-dependent evolution of the thermal strain during the three thermal cycles of the $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam (Fig.3 (a)), the infiltrated MMC $Ni_{60}Nb_{20}Ta_{20}$-AlSi12 (Fig.3 (b)), and for comparison of the AlSi12 matrix itself (Fig. 3 (c)).

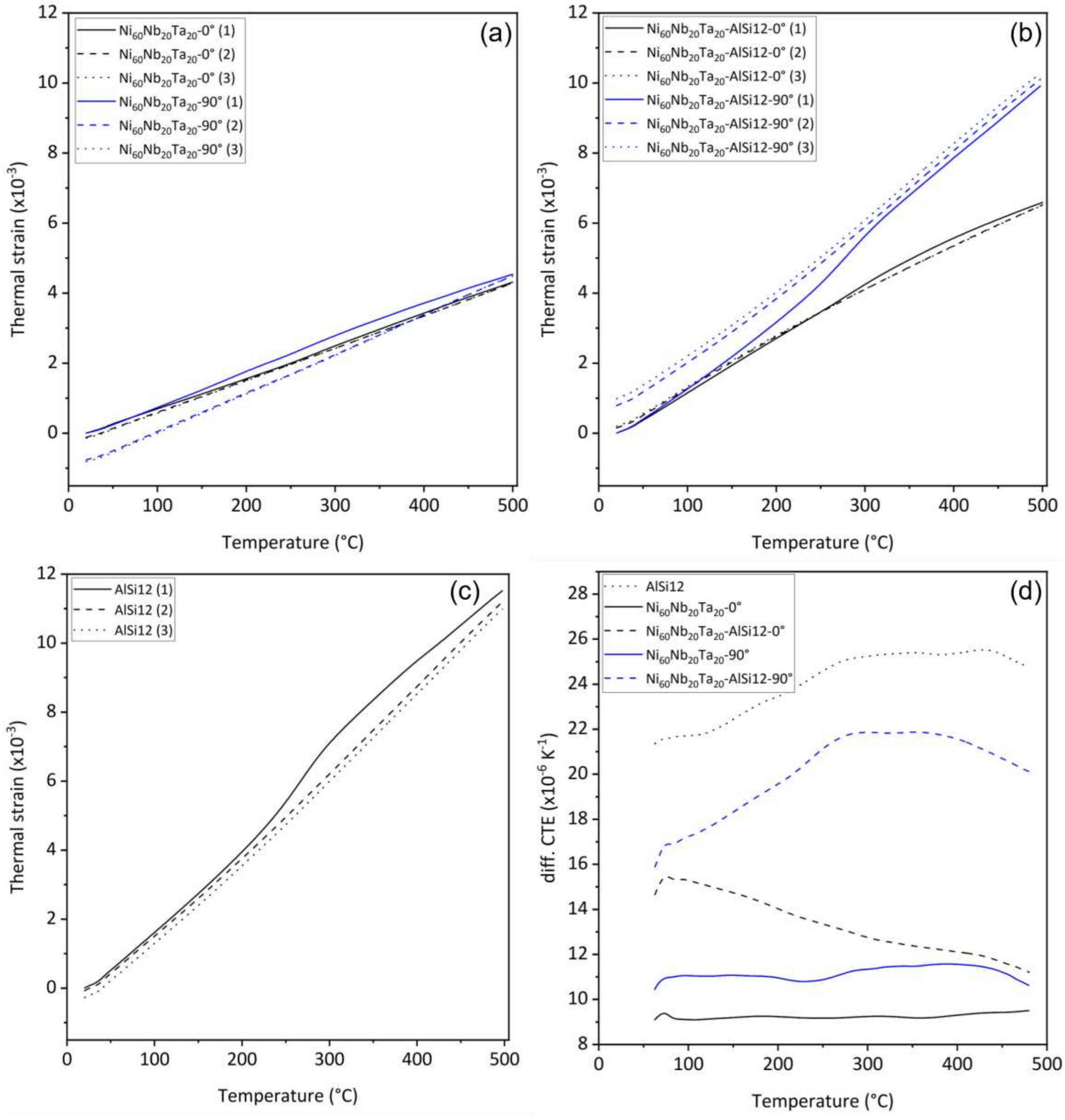

Figure 3. Results of dilatometer measurements. (a) Thermal strain of $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam in 0° and 90° direction. (b) Thermal strain of infiltrated MMC $Ni_{60}Nb_{20}Ta_{20}$-AlSi12 in 0° and 90° direction. (c) Thermal strain of AlSi12. (d) Differential CTE results for all samples.

The first heating cycle of all samples exhibit a distinct increase starting at 200 °C, whereas the following second and third cycle are on the same track. The $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam exhibits with 0.43 % in 0°- and 0.45 % in 90°-direction nearly the same expansion in both directions. However, the sample in 90°-direction shows some remaining negative thermal strain after the cycles, as do the sample of AlSi12. AlSi12 exhibits a max. thermal strain of 1.11 %. The infiltrated MMC combines metallic glass $Ni_{60}Nb_{20}Ta_{20}$ and AlSi12 and results in a max. thermal strain of 0.66 % in 0°-direction and 1.0 % in 90°-direction. Additionally, the samples in 90°-direction exhibit a positive remaining negative thermal strain after the second and third heating cycle. The increase in the first cycle can be attributed to relaxation in the metallic glass [11] and to an internal stress generated in MMCs during solidification at manufacturing process [15]. In order to exclude these influences, only the average value from the second and third heating process is considered in the analysis of CTEs shown in Figure 3 (d). The determined CTE of the metallic glass $Ni_{60}Nb_{20}Ta_{20}$-0° is constant value of $(9.24 \pm 0.10) \times 10^{-6}\,K^{-1}$ with increasing temperature. The CTE of $Ni_{60}Nb_{20}Ta_{20}$-90° remains nearly the same at $(11.17 \pm 0.26) \times 10^{-6}\,K^{-1}$ with a small increase starting at 250 °C. In contrast, the CTEs of the MMC as well as the matrix material AlSi12 are not linear with increasing temperature. The CTE of the $Ni_{60}Nb_{20}Ta_{20}$-AlSi12 in 0°-direction decreases with increasing temperature from $(14.65 \pm 0.19) \times 10^{-6}\,K^{-1}$ at 60 °C to $(11.21 \pm 0.96) \times 10^{-6}\,K^{-1}$ at 480 °C. Whereas, the CTE of the $Ni_{60}Nb_{20}Ta_{20}$-AlSi12-90° is $(15.88 \pm 0.32) \times 10^{-6}\,K^{-1}$ at 60 °C and increases to a maximum of approx. $21 \times 10^{-6}\,K^{-1}$ at 300 °C. Subsequently, the value decreases again to $(20.12 \pm 0.94) \times 10^{-6}\,K^{-1}$ at 480 °C. Similar behavior is observed for the AlSi12-matrix. The CTE starts with $(21.35 \pm 0.25) \times 10^{-6}\,K^{-1}$, increases to a maximum of approx. $25 \times 10^{-6}\,K^{-1}$ between 300 - 400 °C and decreases to $(24.72 \pm 2.88) \times 10^{-6}\,K^{-1}$.

3.2 Specific heat capacity and relaxation

In order to obtain a specific heat capacity independent of the thermal history of the material, an upper temperature limit was first determined experimentally using DSC measurements (Fig. 4 (b)). The DSC signals of the first heating cycle differs from the three following ones.

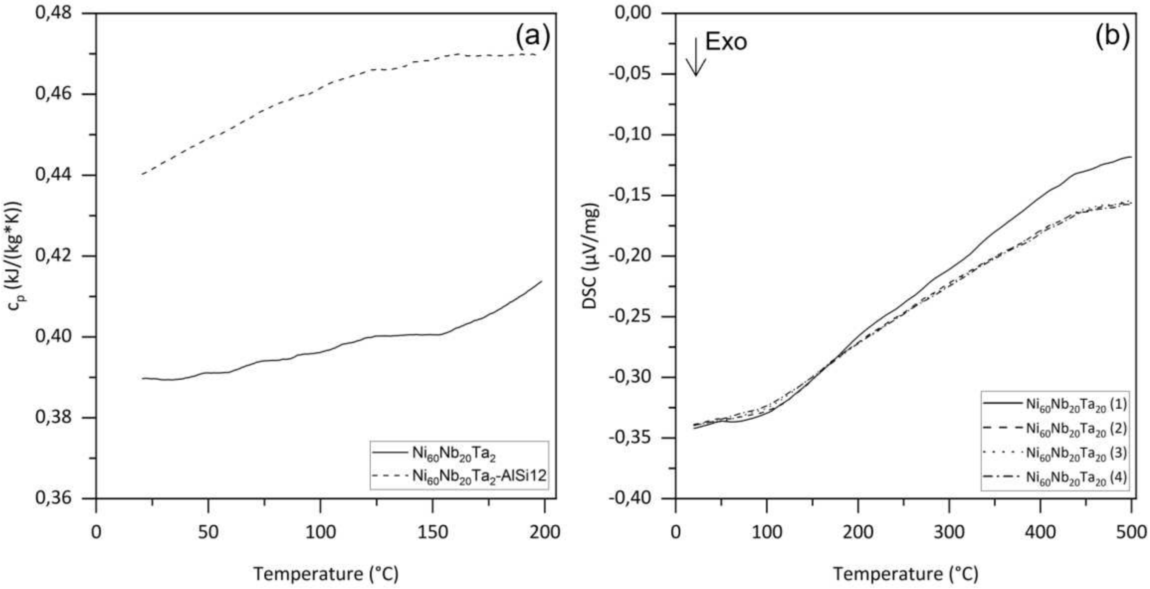

Figure 4. Results of DSC measurements. (a) Specific heat capacity of $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam MMC $Ni_{60}Nb_{20}Ta_{20}$-AlSi12. (b) DSC signal of $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam.

The relaxation temperature T_r therefore corresponds to the temperature at which the DSC signals start to diverge at approximately 200 °C. Consequently, this value was chosen as upper temperature limit for the determination of the specific heat capacity of the metallic glass $Ni_{60}Nb_{20}Ta_{20}$ and the MMC $Ni_{60}Nb_{20}Ta_{20}$-AlSi12 (Fig. 4 (a)). Both c_p values increase with increasing temperature. $Ni_{60}Nb_{20}Ta_{20}$ starts at 0.39 ± 0.01 kJ/kgK at ambient temperature (20 °C) and increases until 0.42 ± 0.01 kJ/kgK at 200 °C. Whereas, the MMC $Ni_{60}Nb_{20}Ta_{20}$-AlSi12 starts with a much higher value at 0.44 ± 0.01 kJ/kgK at 20 °C and increases until 0.47 ± 0.01 kJ/kgK at 200 °C.

3.3 Thermal conductivity

For the evaluation of the AlSi12 sample a density of 2640 kg/m³ and a heat capacity of 0.90 kJ/kgK was used according to literature [16], with a measured thermal diffusivity of $(0.65 \pm 0.02) \times 10^{-4}$ m²/s resulting in a thermal conductivity of 154.43 ± 4.21 W/mK. The thermal conductivity of the infiltrated MMC $Ni_{60}Nb_{20}Ta_{20}$-AlSi12 is 59.44 ± 1.01 W/mK and was calculated with a measured density of 5690 kg/m³, the result of the investigated heat capacity of 0.44 kJ/kgK at ambient temperature and a measured thermal diffusivity of $(0.24 \pm 0.01) \times 10^{-4}$ m²/s. All results are summarized in Table 1.

Table 1. Results of thermal conductivity measured by LFA.

Material	ρ (kg/m³)	$c_{p,20°C}$ (kJ/kgK)	a (x 10^{-4} m²/s)	λ (W/mK)
AlSi12	2640 [8]	0.90 [16]	0.65±0.02	154.43±4.21
$Ni_{60}Nb_{20}Ta_{20}$	10790	0.39±0.01	-	-
$Ni_{60}Nb_{20}Ta_{20}$-AlSi12	5690	0.44±0.01	0.24±0.01	59.44±1.01

4. Discussion

The results show that the thermal expansion of the two components of the MMC differ strongly. Whereas the AlSi12 has a high max. thermal strain of 1.11 % and CTE of 21 to 25 x 10^{-6} K^{-1}, which corresponds with literature [17, 18], the metallic glass $Ni_{60}Nb_{20}Ta_{20}$ exhibits a significantly lower max. thermal strain of 0.43 % and 0.45 % and CTE of 9 to 11 x 10^{-6} K^{-1}. There is no data published for the metallic glass $Ni_{60}Nb_{20}Ta_{20}$ yet, but an estimation according to [19] leads to an approx. CTE of 8.97 x 10^{-6} K^{-1} by using the glass transition temperature $T_g = 936$ K of the same composition in [20], which confirms the results. The metallic glass shows an increase in thermal expansion during the first heating cycle starting at 200 °C, due to thermal relaxation as confirmed by the DSC measurements. In addition, a slight anisotropy between the sample parallel (0°) and perpendicular (90°) to building direction is apparent. AlSi12 also shows an increase during the first heating cycle, starting at 300 °C, which suggests that internal residual stresses already exist in the material. MMC with components whose CTE differ greatly exhibit thermal residual stresses after manufacturing. These are generally expected to be compressive stresses in the reinforcement phase and tensile stresses in the matrix when the CTE of the matrix material is higher than the CTE of the reinforcement phase [15, 21]. All these effects combined lead to the resulting thermal expansion of the MMC $Ni_{60}Nb_{20}Ta_{20}$-AlSi12. Due to the structure of the $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam, the samples exhibit anisotropy in 0°- and 90°-direction. This is further enhanced by the different component proportions in the respective directions. The properties of the metallic glass dominate in 0°- and of the AlSi12 in 90°-direction. Therefore, the MMC in 90°-direction has a significantly higher thermal expansion of 1.0 % than the MMC in 0°-direction with 0.66 %. The anisotropy in the CTE is equally evident. The AlSi12 dominated MMC (90°) behaves similarly to the CTE of AlSi12 and increases from 15 to 21 x 10^{-6} K^{-1}, with increasing temperature. Whereas the metallic glass dominated MMC (0°) decreases linearly from 14 to 11 x 10^{-6} K^{-1}, and thus a linear behavior more typical for metallic glasses [19]. All thermal expansion results are consistent with results of a MMC with $Ni_{60}Nb_{20}Ta_{20}$ as particle reinforcement phase in literature [18].

The resulted specific heat capacity show that AlSI12 with 0.90 kJ/kgK (20 °C) combined with $Ni_{60}Nb_{20}Ta_{20}$ with 0.39 kJ/kgK (20 °C) increases the c_p of the MMC to 0.44 kJ/kgK. The c_p of the metallic glass has not yet been determined, which is why there are no comparative values. However, if the value is compared with the c_p of the individual components ($c_{p,Ni} = 0.45$ kJ/kgK, $c_{p,Nb} = 0.26$ kJ/kgK, $c_{p,Ta} = 0.14$ kJ/kgK [16]), it can be seen that the values correspond well and lead to the conclusion that the results of the MMC also appear realistic.

Same applies to the heat capacity of the MMC, no comparable literature values are yet available. However, the result of the AlSi12 shows that the method provides realistic results, since 154 W/mK corresponds to literature [8, 16]. Considering that nickel (67 W/mK), niobium (54 W/mK) and tantalum (54 W/mK) [16] have a significantly lower thermal conductivity than AlSi12, it can be concluded that the results of the MMC with 59 W/mK appear realistic.

5. Conclusion

A metallic glassy $Ni_{60}Nb_{20}Ta_{20}$ open-porous foam and infiltrated MMC were successfully investigated in terms of thermal expansion, specific heat capacity and thermal conductivity. Thermal expansion was investigated parallel (0°) and perpendicular (90°) to building direction and a pronounced anisotropy was determined. Samples in 90°-direction exhibit a significantly higher thermal expansion with increasing temperature than samples in 0°-direction. The determined heat capacity and thermal conductivity also provide new values for the metallic glass $Ni_{60}Nb_{20}Ta_{20}$ as well as the MMC.

Acknowledgements

The authors are especially thankful to Steffen Czink from the research group "Production and Component Behavior" at the Institute of Applied Materials - Materials Science and Engineering, Karlsruhe Institute of Technology for carrying out the LPBF procedure. Also, special thanks to R. Horny and M. Belli for their technical and experimental support. The financial support of the German Research Foundation (DFG) within the project WE 4273/19-1 is gratefully acknowledged.

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