Rare Metal Materials and Engineering
Volume 54, Issue 11, November 2025
Available online at www.rmme.ac.cn

Science Press

Cite this article as: Yu Zhilei, Li Jingli, Han Xiuzhu, et al. Effect of Temperature on Interface Microstructure and Mechanical Properties of AZ31/Al/Ta Composites Prepared by Vacuum Hot Compression Bonding[J]. Rare Metal Materials and Engineering, 2025, 54(11): 2749-2756. DOI: https://doi.org/10.12442/j.issn.1002-185X.20240604.

ARTICLE

Effect of Temperature on Interface Microstructure and Mechanical Properties of AZ31/Al/Ta Composites Prepared by Vacuum Hot Compression Bonding

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Abstract: AZ31/Al/Ta composites were prepared using the vacuum hot compression bonding (VHCB) method. The effect of hot compressing temperature on the interface microstructure evolution, phase constitution, and shear strength at the interface was investigated. Moreover, the interface bonding mechanisms of the AZ31/Al/Ta composites during the VHCB process were explored. The results demonstrate that as the VHCB temperature increases, the phase composition of the interface between Mg and Al changes from the Mg-Al brittle intermetallic compounds ($Al_{12}Mg_{17}$ and Al_3Mg_2) to the Al-Mg solid solution. Meanwhile, the width of the Al/Ta interface diffusion layer at 450 °C increases compared to that at 400 °C. The shear strengths are 24 and 46 MPa at 400 and 450 °C, respectively. The interfacial bonding mechanism of AZ31/Al/Ta composites involves the coexistence of diffusion and mechanical meshing. Avoiding the formation of brittle phases at the interface can significantly improve interfacial bonding strength.

Key words: AZ31/Al/Ta composites; microstructure; mechanical properties; vacuum hot compression bonding

1 Introduction

Deep-space exploration is a significant indicator of a country's overall national strength and innovation capability. However, as exploration activities venture deeper into space, the materials used for deep-space exploration equipment encounter numerous challenges[1–2]. The high-energy charged particles in the deep-space environment pose a risk of causing radiation damage to electronic components in spacecraft, which can potentially impact exploration missions. Furthermore, the lightweight design is essential for advancing deep-space exploration due to its influence on the carrying capacity of existing spacecraft. Therefore, there is an urgent need to develop a material that combines resistance to energetic particle irradiation with lightweight characteristics to meet the demands of future deep-space exploration.

Currently, metals with high atomic number (Z), such as Nb and Ta, have excellent electron shielding effects and have been used by NASA as high-energy electron shielding materials in the Juno Jupiter probe[3–5]. Moreover, the analysis and calculation outcomes of the anti-radiation material system reveal that, for the same surface density, an optimized combination of low-Z materials (like Mg) and high-Z (like Ta) materials can offer a superior shielding effect compared to a single high-Z material, along with effectively reducing the mass[6–8]. However, the melting points of Ta and Mg are 2996 and 650 °C, respectively. Ta has a body-centered cubic (bcc) crystal structure, and Mg has a hexagonal close-packed (hcp) crystal structure. Due to the significant differences in physical and chemical properties between Mg and Ta, effective diffusion or metallurgical bonding cannot be formed. Therefore, it is relatively difficult to fabricate the Mg-Ta composite. Besides, there are few reports on the preparation of Mg-Ta composite materials worldwide.

Due to the mutual solubility of aluminum (Al) with both magnesium (Mg) and tantalum (Ta), Al can act as a medium

Received date: November 17, 2024
Foundation item: National Natural Science Foundation of China (52275308, 52301146); Fundamental Research Funds for the Central Universities (2023JG007); Supported by Shi Changxu Innovation Center for Advanced Materials (SCXKFJJ202207)
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for joining Mg and Ta, thereby facilitating the preparation of Mg/Ta metal composites. Meanwhile, the rolling method has been utilized to manufacture Mg/Al/Ta multilayer composite plates[9–10]. However, the process involves subjecting Mg, Al, and Ta sheets to differential heat treatment to achieve coordinated deformation during rolling. This includes the oxidation of the sheets and temperature control throughout the rolling process. Consequently, the rolling process requires high-quality equipment and harsh environmental conditions, which leads to low production efficiency due to its complexity. Fortunately, vacuum hot compression bonding (VHCB) offers a new solution to address the aforementioned challenges. However, there are few reports on the fabrication of Mg/Al/Ta layered composite sheets using VHCB method. VHCB is an innovative solid-state bonding technique that utilizes thermo-mechanical coupling to induce significant high-temperature plastic deformation in the interface region, thereby facilitating interface bonding and atomic diffusion, and ultimately achieving metallurgical bonding of the interface[11–12]. Meanwhile, VHCB exhibits outstanding bonding performance and higher bonding efficiency, making it particularly well suited for bonding dissimilar metals and alloys[13–15]. Additionally, the Gleeble thermal simulator has the advantages of rapid heating speed, high heating temperature, and the capability of creating high-pressure and vacuum environments. Therefore, in this study, the optimal VHCB process for Mg/Al/Ta dissimilar metal composites using a Gleeble thermal simulator was investigated.

The AZ31 magnesium alloy is extensively utilized because of its superior strength and ductility compared to pure magnesium. In this work, we aimed to prepare AZ31/Al/Ta dissimilar metal composites by VHCB method. In addition, the feasibility of preparing AZ31/Al/Ta composite materials using VHCB method under different temperature conditions, the microstructure of the interface, and the types and mechanical properties of the secondary phase formed during VHCB process were analyzed. Furthermore, the interfacial bonding mechanism of AZ31/Al/Ta composites was investigated. This would optimize the adaptability of VHCB process and provide a fundamental theoretical basis for preparing AZ31/Al/Ta dissimilar metal composites.

2 Experiment

In this research, commercial AZ31 magnesium alloy sheets, Al foil, and pure Ta sheets were used as raw materials. The chemical composition of these materials is provided in Table 1 – Table 3. The sample sizes are depicted in Fig. 1 (AZ31: Φ16 mm×2 mm, pure Ta: Φ16 mm×2 mm, Al foil: Φ16 mm×0.1 mm). Additionally, stainless steel was employed to fabricate and design the sleeve for assembling and fixing the experimental samples, as shown in Fig. 1. Prior to the experiment, the materials were ground and smoothed using various types of SiC sandpaper. In particular, the surfaces of the Ta and Mg sheets were polished with a copper wheel to enhance their surface roughness. Subsequently, they were immersed in an ethanol solution for ultrasonic cleaning to remove oil stains from their surfaces.

Table 1 Chemical composition of AZ31 alloy (wt%)

Al	Si	Ca	Zn	Fe	Be	Mn	Cu	Mg
3.19	0.02	0.04	0.81	0.005	0.10	0.33	0.05	Bal.

Table 2 Chemical composition of Al foil (wt%)

Si	Zn	Fe	Mn	Cu	Mg	Ti	V	Al
0.25	0.05	0.35	0.03	0.05	0.03	0.03	0.05	Bal.

Table 3 Chemical composition of Ta Sheet (wt%)

C	H	O	Nb	Fe	W	Mo	Si	Ta
0.01	0.015	0.015	0.05	0.005	0.01	0.01	0.005	Bal.

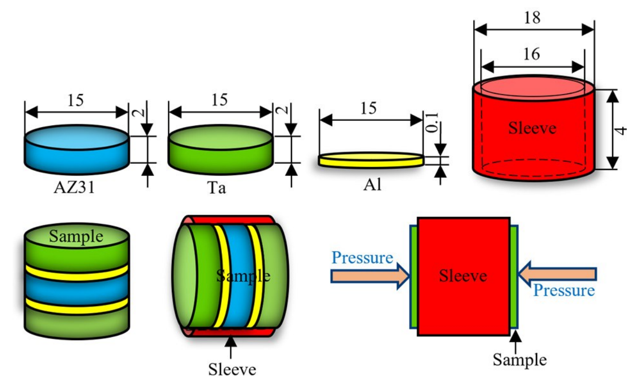

Fig.1 Sample size and assembly diagram

In the experiment, a Gleeble3800 Thermo-Mechanical Simulator (TMS) was used to conduct VHCB on the assembled samples. The schematic diagram of VHCB process using the Gleeble3800 is depicted in Fig.2a. Initially, a k-type thermocouple (TC) with a diameter of 0.25 mm was spot-welded onto the stainless steel sleeve for temperature measurement during the connection process. Subsequently, a small preload was applied to the sample, which was clamped between TMS working pressure heads. TC wire was then connected and the chamber was closed, followed by vacuuming the chamber to 1.33×10^{-2} Pa and initiating the hot compression cycle program. The process parameters are listed in Table 4. During VHCB process, the sample was heated to

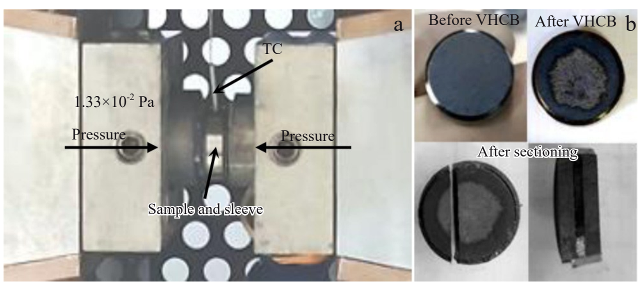

Fig.2 Schematic diagram of VHCB process using Gleeble3800 (a); appearances of samples before and after VHCB and after sectioning (b)

the target temperature at a heating rate of 5 °C/s. The sample was then loaded at a rate of 0.5 MPa/s until the experimental pressure of 50 MPa was reached, which was maintained for 30 min before concluding the VHCB process. Upon completion of the VHCB process, the applied load was rapidly released within 1 min and the sample was subsequently cooled in the ambient air. The appearances of VHCB samples are presented in Fig.2b.

The scanning electron microscope (SEM) observation and energy dispersive spectrometer (EDS) analysis were conducted using a Thermo Scientific Quattro field emission SEM, equipped with an EDS system. The phase composition of the interface was determined by X-ray diffractometer (XRD, Rigaku SmartLab SE). The XRD samples were obtained through the peeling method. Specifically, the Ta layer was removed, and XRD tests were performed on the Mg layer and exposed side of the intermediate layer. Fig. 3 shows the schematic diagram of the measuring method of the shear test. The shear test was performed using a universal testing machine at a loading rate of 0.2 mm/min. At least three samples were tested for each process condition. The fracture surfaces were analyzed using SEM and EDS.

3 Results and Discussion

3.1 Influence of temperature on interlayer thickness

Fig. 4 presents the interlayer thickness changes during the preparation process of AZ31/Al/Ta layered composite materials using VHCB technique under different temperature conditions. Fig. 4a shows SEM image of the sample after VHCB process at 50 MPa/30 min/350 °C. It can be seen that under this process condition, the thickness of the Ta, Al, and AZ31 layers is 2.0, 0.1, and 2.0 mm, respectively, which are the same as the initial ones before VHCB. When the VHCB process temperature increases to 400 °C, the thickness of the

Table 4 Parameters of VHCB process

No.	Holding time/min	Temperature/°C	Pressure/ MPa	Bonding state
1	30	350	50	Unsuccessful
2	30	400	50	Successful
3	30	450	50	Successful

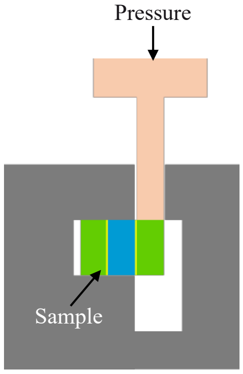

Fig.3 Schematic diagram of shear test measurement method

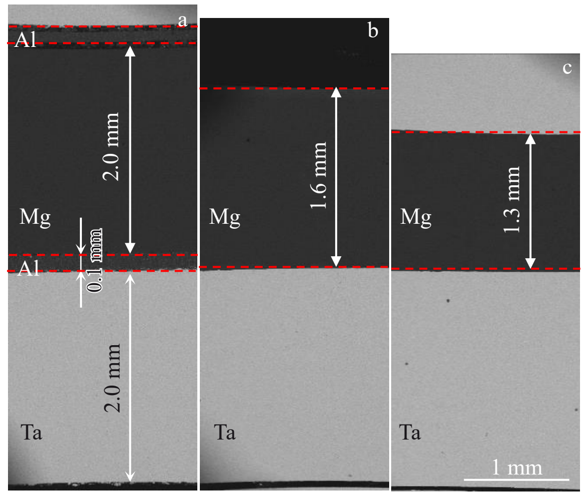

Fig.4 Interlayer thickness of samples prepared by VHCB at different temperatures: (a) 350 °C; (b) 400 °C; (c) 450 °C

Ta layer remains unchanged, while the Al layer is no longer visible, and the thickness of the Mg layer is 1.6 mm. After further increasing the temperature of VHCB process to 450 °C, as illustrated in Fig.4c, it is observed that the thickness of the Mg layer decreases further to 1.3 mm.

During the experiments, it is found that the invisibility of the Al layer and the thinning of the Mg layer are primarily caused by the spillage of molten Mg-Al metal liquid during VHCB process. It is worth noting that VHCB process temperatures mentioned above do not reach the melting points of Mg and Al, whereas a phenomenon of melting still occurs. This is closely related to the heating principle of the Gleeble3800 device. The Gleeble3800 TMS thermal control system works by passing a low-frequency current through the sample, causing heat generation due to electric resistance[15]. In the early stage of VHCB, the sample is clamped with a small preload force, the Mg, Al, and Ta layer materials fail to adhere tightly between the surfaces, and the surfaces touch each other in the form of points, thereby forming a large contact electrical resistance at the contact interface. Therefore, the temperature in the point contact area is significantly higher during the heating process compared to that in other areas, which leads to the overheating and melting of low-melting-point metals Mg and Al, forming a series of microregion liquid phases between the interfaces. Simultaneously, owing to continuous and rapid heating, heat cannot be transferred in time. This leads to interface overheating triggered by microregion overheating, which further leads to more Mg and Al melting. After the short-term rapid heating is completed, a target pressure of 50 MPa is quickly applied. During the process of pressure loading, the molten Mg and Al liquid phases are squeezed and spilled out, resulting in a reduction in interlayer thickness.

3.2 Interfacial structure and chemical composition

SEM images of the interface of AZ31/Al/Ta samples after VHCB at different temperatures are shown in Fig. 5. Fig. 5a

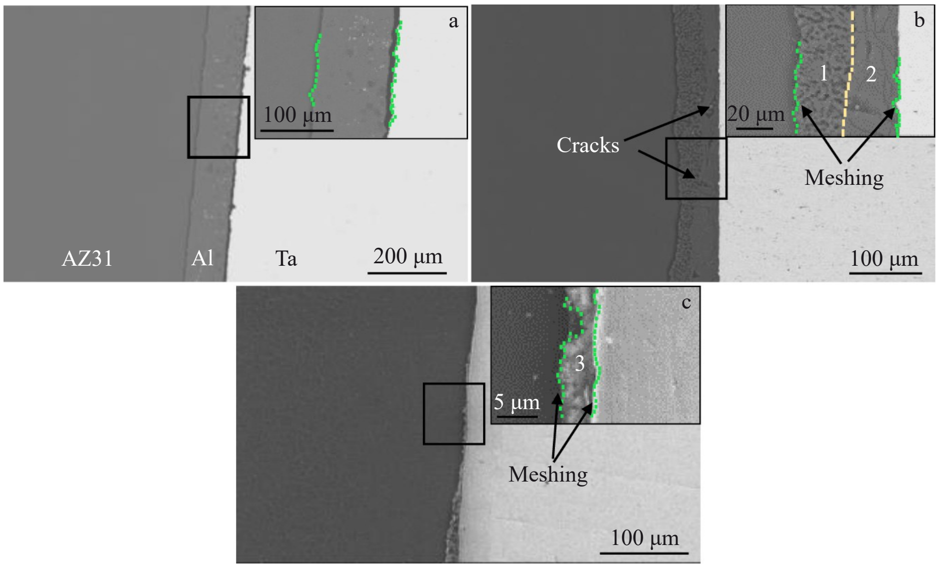

Fig.5 SEM images of interface of AZ31/Al/Ta samples after VHCB at 350 °C (a), 400 °C (b), and 450 °C (c)

shows the interface of the sample prepared under the conditions of 50 MPa/30 min/350 °C. It can be seen that the Mg/Al interface tightly contacts and binds under bonding pressure, without the formation of intermetallic compounds (IMCs) at the interface. The Al/Ta interface spalls during the cutting owing to its low bonding strength. It can also be seen from Fig. 5a that a meshing shape is formed at the Al/Ta interface under pressure.

As depicted in Fig.5b, effective bonding is achieved at both Mg/Al and Al/Ta interfaces when VHCB temperature reaches 400 °C. Moreover, it also shows that the interlayer thickness decreases to approximately 60 μm. Simultaneously, a significant change in the morphology of the interlayer structure is observed, displaying distinct features on the side near Mg and Ta. Therefore, EDS analysis is conducted at positions 1 and 2, as shown in Fig.5b. Table 5 presents the chemical composition at various positions. A layer with a thickness of 10–30 μm can be formed close to the Mg side, which exhibits a stable stoichiometric proportion of Mg to Al close to 17:12, indicating that this layer is composed of $Al_{12}Mg_{17}$ (γ)[16–18]. Similarly, adjacent to position 2, the content of Al and Mg is close to 3:2, suggesting that position 2 is composed of Al_3Mg_2 (β)[17–18]. Further observations show that there are obvious microcracks in the formed IMCs. This is due to the plastic deformation of the composite material caused by the combined effect of pressure and temperature during the VHCB process. It is well known that IMCs have far higher hardness than matrix materials. Hence, plastic incompatibility during the VHCB process may result in cracks within the interlayer of hard and brittle IMCs[19–20]. This also implies that the presence of such large brittle IMCs has a detrimental influence on the interfacial structure. Additionally, Fig. 5b illustrates that a meshing shape is formed at the interface, indicating that a mechanical meshing phenomenon also appears at the interface.

Table 5 EDS results of positions marked in Fig.5

Position	Element	wt%	at%	Uncertain content/%
1	Mg	58.1	60.6	1.9
	Al	41.9	39.4	8.8
2	Mg	37.5	40.0	2.2
	Al	62.5	60.0	7.1
3	Mg	20.8	22.5	4.2
	Al	79.2	77.5	6.1

When VHCB temperature is further increased to 450 °C, effective bonding is also achieved at the Mg/Al and Al/Ta interfaces, as shown in Fig. 5c. However, compared to the condition at 400 °C, the thickness of the interlayer is significantly decreased. EDS analysis is also performed at position 3 (Fig.5c). The results show that the contents of Mg and Al are 22.5at% and 77.5at%, respectively, as shown in Table 5. This indicates that no brittle IMCs are formed under the process conditions of 450 °C. It is worth noting that the presence of a meshing shape at the interface is also observed at 450 °C.

The above results indicate that no Ta is detected at the interface of the intermediate layer. This can be attributed to the high diffusion activation energy required for the migration of Ta, which cannot be achieved under the aforementioned temperature conditions. Consequently, the Ta atoms do not diffuse to the interface.

Furthermore, it is noteworthy that a mechanical meshing phenomenon is observed at the interface in the aforementioned results, and previous studies have indicated that similar phenomena are also detected in rolling and explosive welding processes[21–23]. To enhance the mechanical properties of explosive-welded Al-Fe transition joints, Yang et al[22–23] obtained the meshing interface by prefabricating dovetail grooves on the base plate. Similarly, in order to enhance the interfacial bonding between Al and Ta, the surfaces of the Ta plate and Mg plate are roughened prior to thermal compression in this study. Therefore, during the

VHCB process, when the interfacial layer undergoes melting, the liquid metal phase will fill the irregular surfaces of Ta and Mg plates due to thermal-mechanical coupling effects, thereby forming a more microscopic mechanical meshing interface.

The analysis of SEM results above reveals that the secondary phase is only observed at the Mg/Al interface. To further clarify the phase composition of the interface during the VHCB process, XRD analysis is performed on the Mg/Al interface of the samples (400 and 450 °C), as shown in Fig.6. At 450 °C, no secondary phase is detected at the Mg/Al interface, whereas at 400 °C, the formation of Al_3Mg_2 and $Al_{12}Mg_{17}$ phases is evident. This further validates the accuracy of the above findings.

Fig. 7 shows SEM images and corresponding XRD line scanning results under various temperature conditions. The elemental distribution of the Mg/Al bonding interface prepared at 350 °C is shown in Fig.7a. It can be observed that the Mg/Al interface diffusion layer thickness is approximately 10.3 μm. As shown in Fig.7b, when the temperature increases to 400 °C, the thickness of the diffusion layer formed between Mg/Al and Al/Ta is approximately 69.5 and 1.1 μm, respectively. Furthermore, when VHCB temperature is increased to

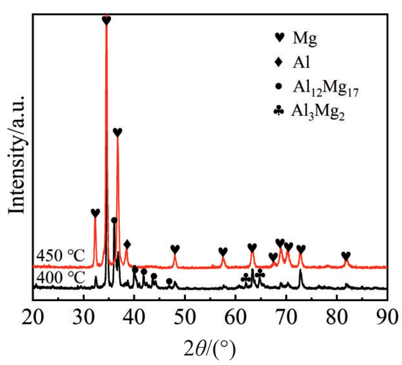

Fig.6 XRD results of interfacial secondary phase of samples prepared at 400 and 450 °C

450 °C, as illustrated in Fig. 7c, it can be observed that the thickness of the diffusion layer formed between Mg/Al and Al/Ta interfaces is approximately 9.0 and 2.3 μm, respectively.

The previously mentioned findings demonstrate that temperature significantly influences the interface structure and chemical composition. An appropriate temperature is an important parameter of VHCB process[24–25]. Based on the above discussion, it can be concluded that as the temperature increases from 350 °C to 450 °C, both the thicknesses of the diffusion layer and interface structure show significant differences. At 350 °C, the bonding mechanism at the Mg/Al interface involves a combination of solid-state diffusion and mechanical meshing. The above findings also indicate weak mechanical meshing at the Al/Ta interface before peeling. When the temperature increases to 400 and 450 °C, both Mg/Al and Al/Ta interfacial bonding mechanisms are a combination of diffusion and mechanical meshing. The interface structures and chemical composition of AZ31/Al/Ta composites fabricated using VHCB technique at 400 and 450 °C are compared. A significant number of brittle IMCs ($Al_{12}Mg_{17}$ and Al_3Mg_2) are formed at the interface under the processing conditions of 400 °C. In contrast, no brittle phases are observed at the interface under the processing conditions of 450 °C. According to the Mg-Al binary phase diagram in the Mg-Al binary system, the liquid phase appears at crystal melting temperature T_m (T_m=437 °C). Meanwhile, at the process temperature of 400 °C, accompanied by the elimination of overheating, the supersaturated Mg-Al liquid phase at the interface satisfies the thermodynamic conditions for the precipitation of Mg-Al IMCs during the cooling process to 400 °C ($T<T_m$). Therefore, IMCs are easily nucleated under these conditions. With the prolongation of holding time, IMCs gradually increase in size, eventually leading to the formation of large-sized γ and β IMCs. On the contrary, under the process condition of 450 °C, Mg-Al IMCs do not possess nucleation possibility during the process of interface cooling

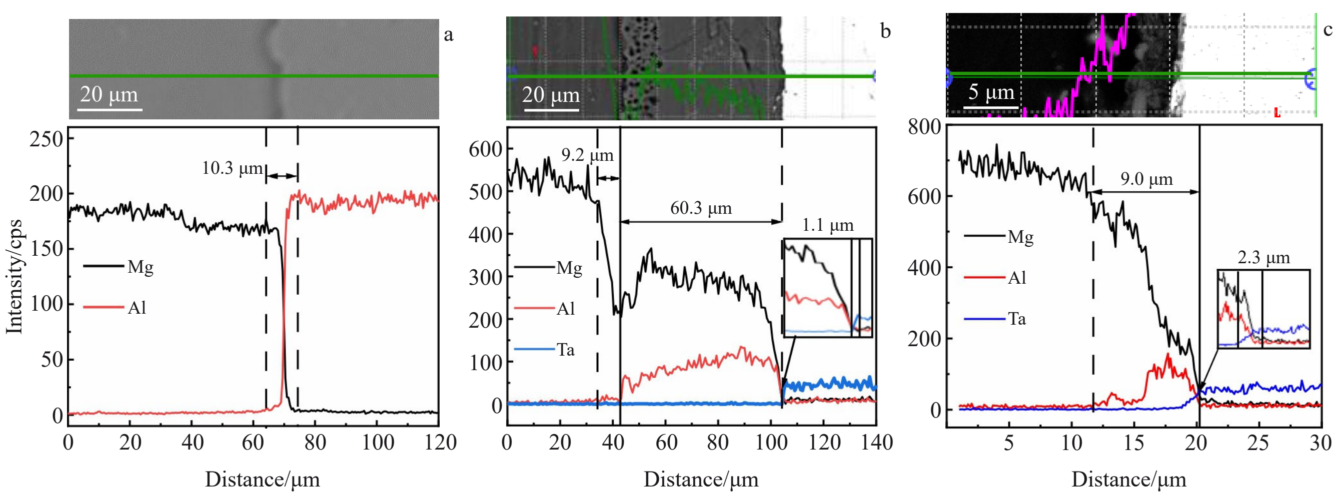

Fig.7 SEM images and corrsponding XRD line scanning results of Mg/Al bonding interface prepared at different temperatures: (a) 350 °C; (b) 400 °C; (c) 450 °C

from overheated temperature to 450 °C ($T>T_m$). The interface remains in the form of a eutectic liquid phase during this process, and in the subsequent long-term holding process, the interface elements continue to diffuse and undergo isothermal solidification. Under this temperature process condition, the holding stage after solidification also has the advantage of homogenization treatment, which can minimize element segregation and reduce the probability of generating Mg-Al IMCs to the maximum extent[26]. Furthermore, compared to that at 400 °C, the width of the Al/Ta interface diffusion layer increases at 450 °C. Overall, it can be assumed that the preparation of AZ31/Al/Ta composites by VHCB technique at 450 °C can yield an excellent interface structure.

3.3 Mechanical properties and fracture behavior of interface

The room-temperature shear strengths of the samples under different temperature conditions are shown in Table 6. The shear strengths are 24 and 46 MPa at 400 and 450 °C, respectively. Compared with the process conditions at 400 °C, the shear strength of the AZ31/Al/Ta composite material is significantly improved at 450 °C.

The shear fracture surfaces on the Ta side of the samples at 400 and 450 °C are shown in Fig. 8. The corresponding elemental composition analysis of the fracture surface is presented in Table 7. It shows the Ta matrix and Al_3Mg_2 phase, as indicated by the arrows in Fig.8a–8b. This implies that the fracture of the bonded joints occurs mainly at the interface between the Al_3Mg_2 phase and Ta connection. Meanwhile, there is a large amount of parallel striation on the fracture surface, which can be determined as brittle fracture characteristics[27]. The fracture of Mg-Al brittle IMCs may become the main cause of crack propagation and interface cracking[28]. The aforementioned results further suggest that the interfacial strength between the brittle phase and Ta is relatively weak. However, as shown in Fig.8c–8d, the fracture surface at 450 °C is mainly composed of a small amount of Ta and a large amount of Mg-Al alloy layer. This also implies that the fracture of the bonded joints occurs mainly on the interface between the Mg-Al alloy layer. This further indicates that the interfacial strength between the Mg-Al layer and Ta is relatively strong.

Table 6 Shear strength of samples under condition of 50 MPa/30 min at different temperatures

Temperature/°C	Shear strength/MPa
400	24
450	46

3.4 Technical principle of VHCB for preparation of AZ31/Al/Ta composites

Based on the above discussion and phase diagram analysis, the interface structure evolution model of the AZ31/Al/Ta composites prepared by VHCB technique can be obtained, as shown in Fig.9. Firstly, as shown in Fig.9a, in the early stage of VHCB, due to the unevenness of the material surface, there is a point contact phenomenon at the interface, which leads to microregion overheating during the process of Gleeble heating to the target temperature ($T>T_m$). Therefore, several VHCB microregions are formed at the interface. Secondly, Fig. 9b shows that during the continuous heating process, microregion overheating triggers interface overheating, leading to the melting of Mg and Al and the formation of a large liquid-phase zone. Due to thermo-mechanical coupling, the Mg and Al liquid phases diffuse and fuse, while atomic diffusion occurs at the Al/Ta liquid-solid interface. As the pressure increases, the Mg and Al liquid phases fill the interface gaps, and the overheating phenomenon gradually subsides. During this period, the element Al continuously diffuses from the liquid

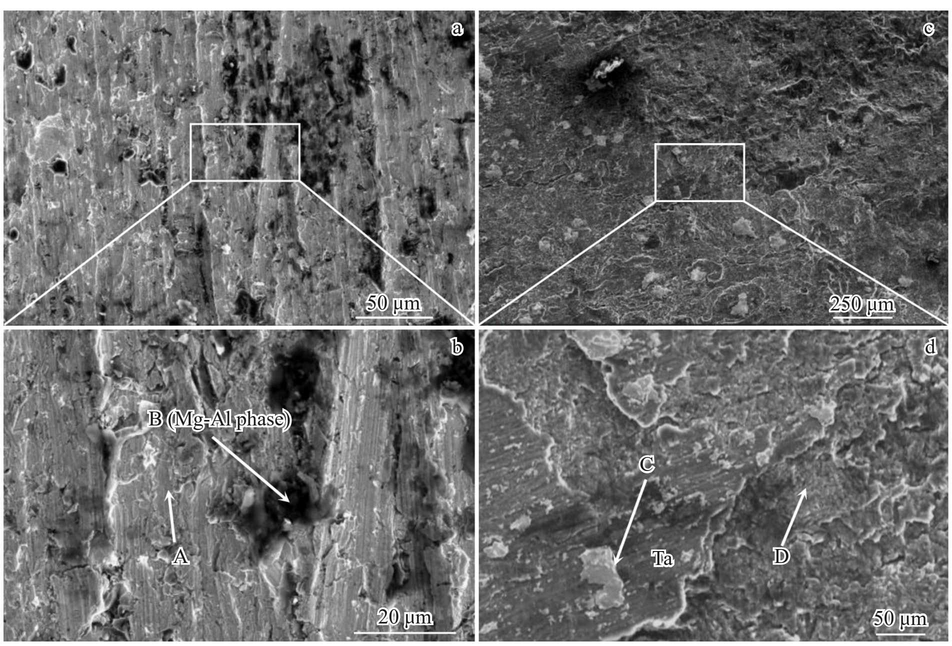

Fig.8 SEM images of fracture surfaces at Ta side of samples prepared at different temperatures: (a–b) 400 °C; (c–d) 450 °C

Table 7 Elemental composition analysis of positions marked in Fig.8b and 8d

Position	Element	wt%	at%	Uncertain content/%
A	Mg	2.9	16.4	12.6
	Al	2.2	11.1	13.0
	Ta	95.0	72.5	7.3
B	Mg	38.4	40.9	2.4
	Al	61.6	59.1	6.7
C	Ta	100.0	100.0	7.0
D	Mg	25.2	56.3	6.1
	Al	12.4	24.9	8.7
	Ta	62.5	18.8	9.2

phase into both sides of the base metal, further increasing the width of the liquid phase until the concentration of elements in the base metal and liquid phase reaches equilibrium in the phase diagram at the interface. Thirdly, according to the lever law of equilibrium solidification of the binary alloy phase diagram, as the alloy elements and base metal elements in the liquid phase diffuse into each other, the volume of the liquid phase gradually decreases, and isothermal solidification occurs during this process, as shown in Fig.9c. Accompanied by the process of solidification, a meshing shape begins to form at the interface under thermal-mechanical coupling. Finally, as shown in Fig. 9d, there is still an uneven distribution of alloying elements after isothermal solidification. During the subsequent holding stage, the

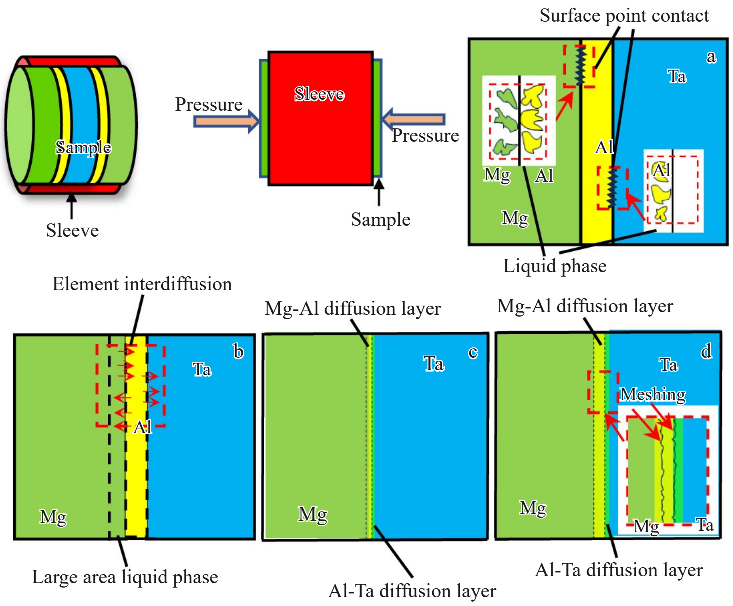

Fig.9 Technical principle of VHCB for preparation of AZ31/Al/Ta composites

alloying elements undergo solid-phase diffusion, which leads to an increase in width of the diffusion layer. This process further reduces element segregation and ultimately achieves a uniform interfacial element concentration

Thus, from the above discussion, the AZ31/Al/Ta composites with good interfacial bonding properties can be obtained at a VHCB temperature of 450 °C ($T>T_m$). Under this process condition, the AZ31/Al/Ta interface has uniform microstructure without forming a brittle secondary phase, creating a bonding interface where diffusion and mechanical meshing coexist.

4 Conclusions

1) The AZ31/Al/Ta composites are prepared using VHCB method with process parameters of 50 MPa/30 min/400 °C and 50 MPa/30 min/450 °C.

2) The interfacial microstructure is significantly affected by temperature. Under the processing condition of 400 °C, a considerable number of brittle IMCs ($Al_{12}Mg_{17}$ and Al_3Mg_2) form at the interface. In contrast, no brittle phases are formed at the interface under the processing condition of 450 °C.

3) The shear strengths are 24 and 46 MPa at 400 and 450 °C, respectively. VHCB temperature at 450 °C can lead to high shear strength because it avoids the formation of brittle phase at the interface.

4) The interface bonding mechanism of the AZ31/Al/Ta composite fabricated via VHCB method at 450 °C is characterized by a synergistic combination of diffusion and mechanical meshing.

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温度对真空热压连接技术制备AZ31/Al/Ta复合材料界面组织和力学性能的影响

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摘 要： 采用真空热压连接方法制备了AZ31/Al/Ta复合材料。研究了热压温度对AZ31/Al/Ta复合材料界面组织演变、相组成及界面抗剪切强度的影响。探讨了AZ31/Al/Ta复合材料在真空热压连接工艺条件下的界面结合机理。结果表明，随着热压温度的升高，Mg-Al界面相组成由Mg-Al脆性金属间化合物（$Al_{12}Mg_{17}$和Al_3Mg_2）转变为Al-Mg固溶体。与400 ℃相比，450 ℃时Al/Ta界面扩散层宽度增大。在400和450 ℃时，AZ31/Al/Ta复合材料抗剪切强度分别为24和46 MPa。AZ31/Al/Ta复合材料界面结合机制为扩散与机械啮合共存。此外，避免界面脆性相的形成可以显著提高界面结合强度。

关键词： AZ31/Al/Ta复合材料；微观组织；力学性能；真空热压连接

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