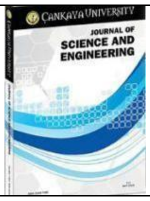
e-ISSN: 2564-7954 CUSJE 22(1): 016-022 (2025) Research Article



Çankaya University

Journal of Science and Engineering

https://dergipark.org.tr/cankujse

CUJSE

Wettability of Mg and AZ91 Alloy on Ti6Al4V Alloy Substrate

Ezgi Bütev Öcal[1*] Arcan F. Dericioğlu[1]

[1] Metallurgical and Materials Engineering Department, Middle East Technical University, Ankara / Türkiye

Keywords

Wettability,
Magnesium alloys,
Ti6Al4V alloy,
Scanning electron microscope.

Abstract

The wettability of liquid Mg and AZ91 alloy on bulk Ti6Al4V alloy was investigated in the current study. Wetting experiments were conducted by gradually heating the samples to 800°C for 30 minutes. A CCD camera was used to capture the morphological changes in the droplet during contact angle measurements. Additionally, a scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS) was used to analyze the morphology and microstructure of the wetted surfaces of the Ti6Al4V alloy. X-ray diffraction analysis (XRD) was performed so as to examine the phase constituents in the reaction layer of the Ti-alloy substrate and magnesium and AZ91 alloy.

1. Introduction

The manufacturing of titanium-magnesium composites, which are highly desirable because of their exceptional mechanical properties and their uses in lightweight structural designs, depends mainly on the wetting behavior of liquid magnesium on titanium alloys. The capacity of a liquid to spread across a solid surface is known as wetting. It directly affects the interfacial interactions between titanium and magnesium, which in turn affects the microstructure and bonding strength of the resulting composite materials [1]. Optimizing titanium-magnesium composites' manufacturing processes and enhancing their performance in a variety of industries, including the biomedical, aerospace, automotive, and energy sectors, require an extensive understanding of these wetting properties [2].

The limitations of conventional metallic biomaterials are intended to be addressed by the production of innovative functional metal-metal composites. In order to preserve mechanical stability, degradable metals are frequently mixed with non-degradable metals because they lose their structural integrity as they corrode [3]. For example, titanium-magnesium bioactive composite produced by hot extrusion demonstrated fatigue limits that were appropriate for dental implants [4]. According to in vitro tests, magnesium corroded with time and increased surface roughness. The findings revealed that these bioactive composites could be used as dental implants because they provide improved bioactivity and mechanical compatibility. Moreover, Jiang et al. used the infiltration casting approach to create a magnesium-based composite by infiltrating molten magnesium into a porous titanium framework [5]. According to their research, these composites' mechanical properties were similar to those of cortical bone, indicating that titanium-magnesium composites hold a lot of promise for load-bearing biomedical applications [5]. Furthermore, Ti6Al4V-Mg and Ti6Al4V-AZ91 composites were manufactured by Esen et al.

---

* Ezgi Bütev Öcal: butevezgi@gmail.com

Received: March 23, 2025, Accepted: April 10, 2025

for use in biomedical applications [6]. The Ti6Al4V-Mg composite failed as a result of galvanic corrosion caused by the combination of unalloyed magnesium and Ti6Al4V. However, because of the AZ91 alloy's low rate of corrosion and the formation of $TiAl_3$ at the interface layer, Ti6Al4V-AZ91 composites maintained their mechanical integrity.

In order to better understand the interface formation behavior, which is essential for the design and optimization of the manufacturing of titanium–magnesium composites, the wettability of magnesium on titanium alloy is examined in the present study. The presence of intermetallic phases at the interface between Mg and AZ91 alloy and the Ti6Al4V substrate is investigated, and the change in contact angle is measured.

2. Materials and Method

Bulk Ti6Al4V alloy bars with a diameter of 5.5 mm were obtained from Philly Materials. The chemical composition of titanium alloys meeting the ASTM F 1580-01 standard [7] consists of 6.20% Aluminum (Al), 4.10% Vanadium (V), 0.06% Iron (Fe), 0.01% Carbon (C), 0.004% Hydrogen (H), 0.1% Nitrogen (N), 0.1% Oxygen (O), and balance Ti in wt.%.

As-cast bulk samples of pure magnesium and AZ91 alloy were sourced from the Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, China. Pure Mg consists of 0.025% Al, 0.057% Zinc (Zn), 0.071% Manganese (Mn), 0.027% Neodimyum (Nd), 0.0356% Copper (Cu), 0.0176% Fe, 0.072% Nickel (Ni), 0.035% Silicon (Si), and 99.50% Mg, in wt.%, and AZ91 alloy contains 9.0% Al, 0.8% Zn, 0.2% Mn, and 90% Mg in wt.%.

Magnesium's wettability and any possible chemical interactions at the interface between liquid magnesium and titanium alloy substrates were evaluated using contact angle measurements. An atmosphere-controlled tube furnace was built for the wetting studies. The Ti6Al4V alloy substrate was 16 mm in diameter and 4 mm in height, whilst the bulk Mg and AZ91 alloys were 6 mm in diameter and 4 mm in height. The samples were heated in a high-purity argon (Ar) gas atmosphere at a rate of 10°C/min until they reached 800°C. During the contact angle measurements, the morphological changes in the droplet were observed using a CCD camera. The morphology and microstructure of the wetted surfaces of the Ti6Al4V alloy were analyzed using a scanning electron microscope (SEM), FEI 430 Nano Scanning Electron Microscope, equipped with energy-dispersive X-ray spectroscopy (EDS). The surface roughness of the Ti6Al4V alloy was investigated via Atomic Force Microscope (AFM, Veeco MultiMode V) in tapping mode. X-ray diffractometer (XRD) measurements were carried out using a Rigaku D/Max 2200/PC (Tokyo, Japan) equipped with Cu Kα radiation, at 30 mA current and 40 kV voltage. The scanning rate is 2°/min between 10° to 90°.

3. Results

The morphological change of magnesium on the Ti6Al4V substrate at different exposure times to 800°C is given in Figure 1. During the first five minutes (t2) of the experiment, there was no observable change in the magnesium's shape. Within seconds, the centers of the sample rapidly collapsed inward after melting at the center (t3). Then, at t6, the whole magnesium quickly wetted the surface of Ti6Al4V. It is known that up to about 450°C, magnesium oxidizes quite slowly; after that, oxidation speeds up considerably, according to Kumar et al. [8]. In the current study, the reason for the observed delay in melting could be the development of an oxide layer on the solid magnesium. Before the molten magnesium completely spread throughout the titanium surface (t8), the outer shell, which likely consisted of magnesium oxide, stayed unaffected for a while (from t3 to t6), even though the samples started to collapse from the center, in Figure 1.

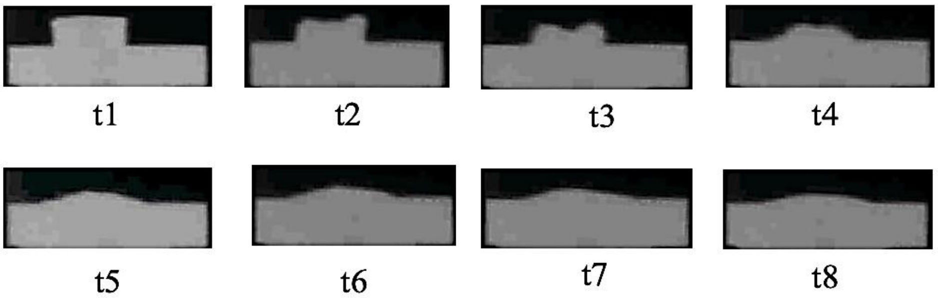


Figure 1. Shape changes of Mg in titanium alloy substrate at different time intervals.
(t1: 0 min., t2: 5.5 min., t3: 5.58 min., t4: 6.02 min., t5: 6.07 min., t6: 6.12 min., t7: 7.45 min., t8: 8.09 min.)

Three separate stages, shown in Figure 2, represent the variation in contact angles of liquid magnesium. The wetting angle stayed almost constant for the first five minutes, as also observed in Figure 1. Kondoh et al. [9] reported a similar observation. According to their explanation, the early stability of the contact angle was caused by liquid magnesium reducing the native TiO_2 layer on pure titanium [9–10]. Within a minute, the contact angle in the second stage fell abruptly to about 12°, as shown in Figure 2. Following that it stayed steady following this abrupt drop. The unexpected flow of liquid magnesium was attributed to this dramatic drop as a result of the cracking of the oxide layer surrounding the molten metal [9,11]. The titanium alloy substrate was almost completely wetted as the liquid magnesium spread. Nevertheless, as already shown in earlier research, the contact angles are directly impacted by the sudden evaporation of pure magnesium [9–11].

Moreover, the diameter of pure magnesium remained steady during the early stage, aligning with a stable contact angle. However, as soon as the contact angle started to change suddenly, a sharp rise in diameter was seen in Figure 2. It is also stated that pure magnesium's high vapor pressure at 800°C can cause the molten droplet to expand when it evaporates at high temperatures [9]. In line with these results, the current investigation found significant variations in wetting behavior and droplet size that occurred when magnesium melted and evaporated simultaneously.

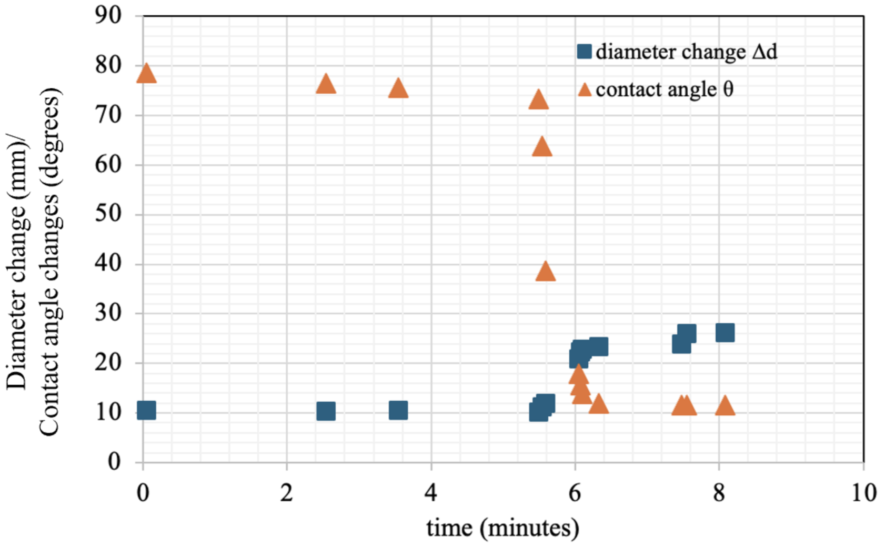
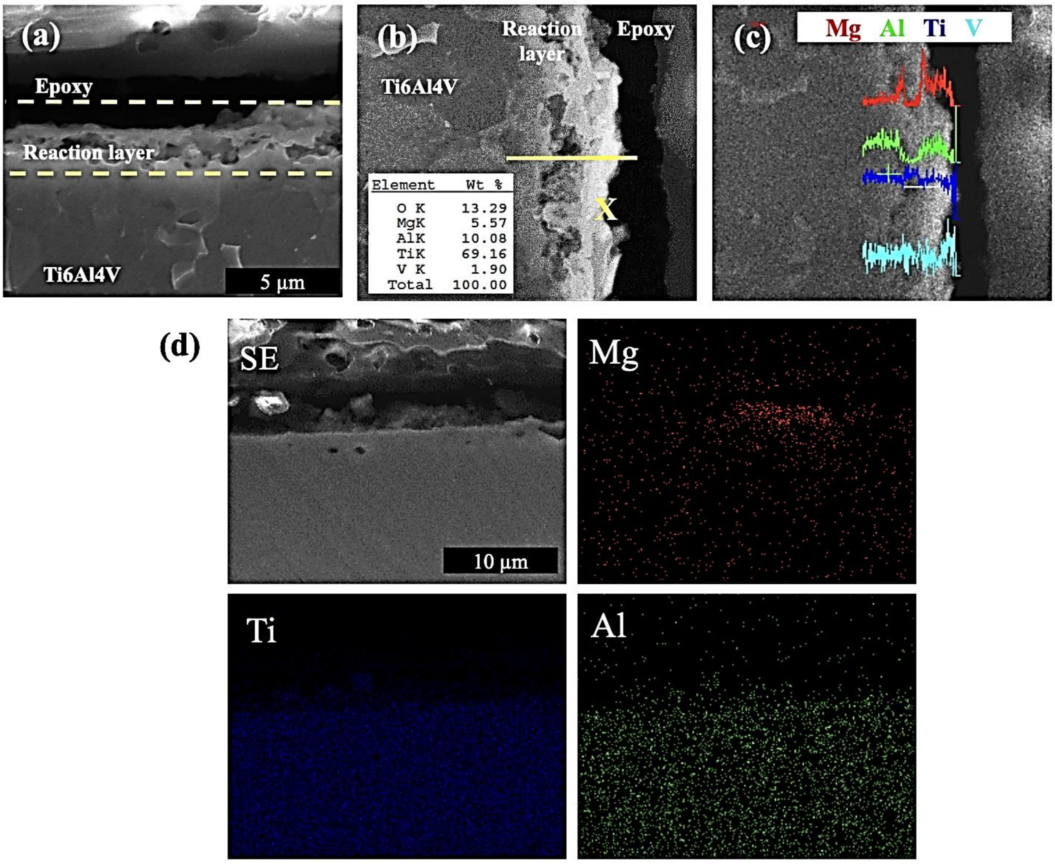


Figure 2. The change in the diameter and the contact angle of M at 800°C.

It is known that the substrate's surface condition has a direct impact on the liquid's wetting behavior [12]. Thus, the average surface roughness of the starting Ti6Al4V alloy was measured as 146 ± 40 nm by AFM. SEM pictures of the reaction layer that forms on solid Ti6Al4V alloy when it comes into contact with liquid magnesium are shown in Figure 3. This layer was 2–5 μm thick and showed a porous texture. EDS spot analysis revealed that its main constituents were 13.40 wt.% O and 5.6 wt.% Mg. These components' presence on the Ti-alloy surface supports the theory that MgO is formed when liquid magnesium reduces TiO_2. Only α-Ti and the titanium oxide phase, TiO, which was most likely generated during the cooling stage, were found in the reaction layers according to XRD analysis, Figure 4(a).



Element	Wt %
O K	13.29
MgK	5.57
AlK	10.08
TiK	69.16
V K	1.90
Total	100.00

Figure 3. (a) SEM and (b) EDS spot analysis shown by X, (c)EDS line, and (d) EDS map scan analysis of reaction layer.

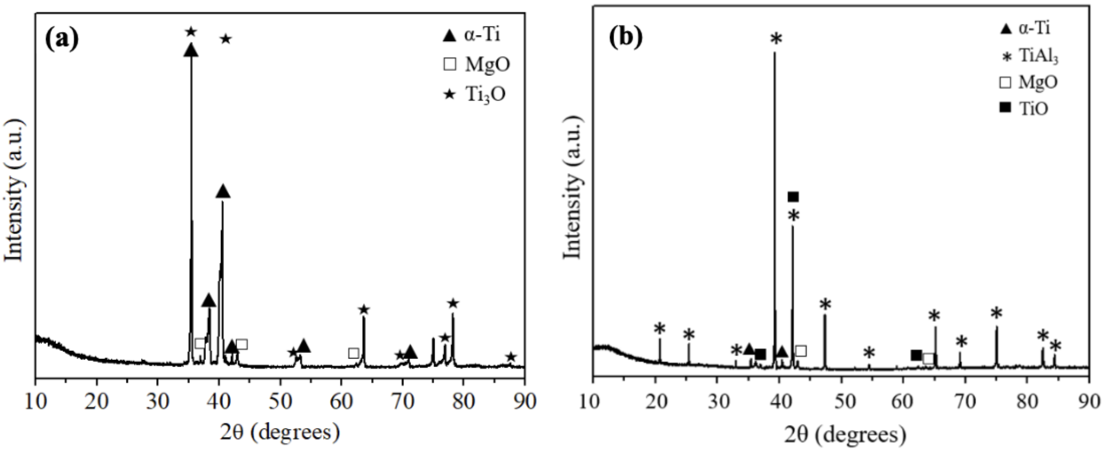
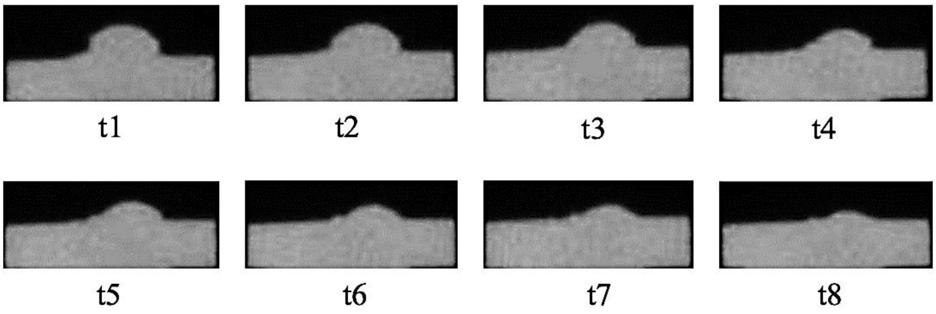


Figure 4. XRD results of titanium alloy substrate wetted by (a) Mg, and (b) AZ91 alloy.

The structure change of the AZ91 alloy on the Ti6Al4V substrate during the soaking test is depicted in Figure 5. The moment AZ91 alloy began to melt was taken as the reference point (t1=0 minutes). The Ti6Al4V surface was evenly wetted by the liquid AZ91 alloy after 22 minutes. The contact angle between the liquid AZ91 alloy and the Ti6Al4V alloy progressively reduced in comparison with pure magnesium. According to Q. Tan et al., Zn lowers the oxidation resistance of magnesium alloys, and substantial magnesium evaporation may result from the existence of Mg-Zn intermetallic phases with low melting temperatures [13]. They also stated that Zn tends to sublime, which speeds up the cracking of the oxide layer. In the current study, because of the broken oxide layer that covers liquid magnesium, the melt pool of AZ91 alloy emerged earlier in the wetting experiment compared to pure Mg.

Figure 6 depicts the measured wetting angle values after the AZ91 alloy had melted completely. The liquid AZ91 alloy's contact angle was decreasing over time. The liquid AZ91 droplet wetted the Ti6Al4V surface after 22 minutes at a contact angle of around 12°, which is close to that of pure magnesium. Yet in contrast to pure magnesium, the melt pool diameter contracted, likely due to the higher evaporation rate of magnesium caused by the presence of Zn.


t1 t2 t3 t4

t5 t6 t7 t8

Figure 5. Shape changes of AZ91 alloy in titanium alloy substrate at different time intervals. (t1: 0 min., t2: 5 min., t3: 10 min., t4: 12 min., t5: 14 min., t6: 18 min., t7: 20 min., t8: 22 min.)

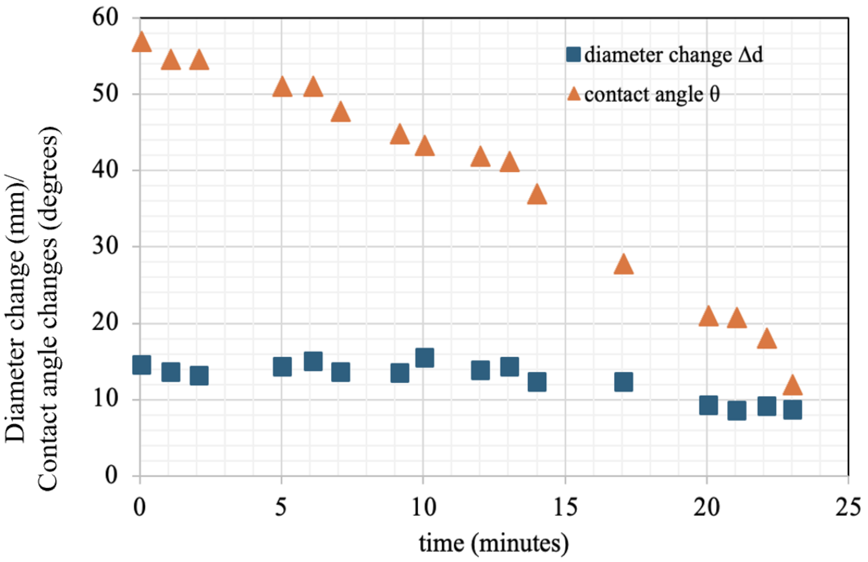
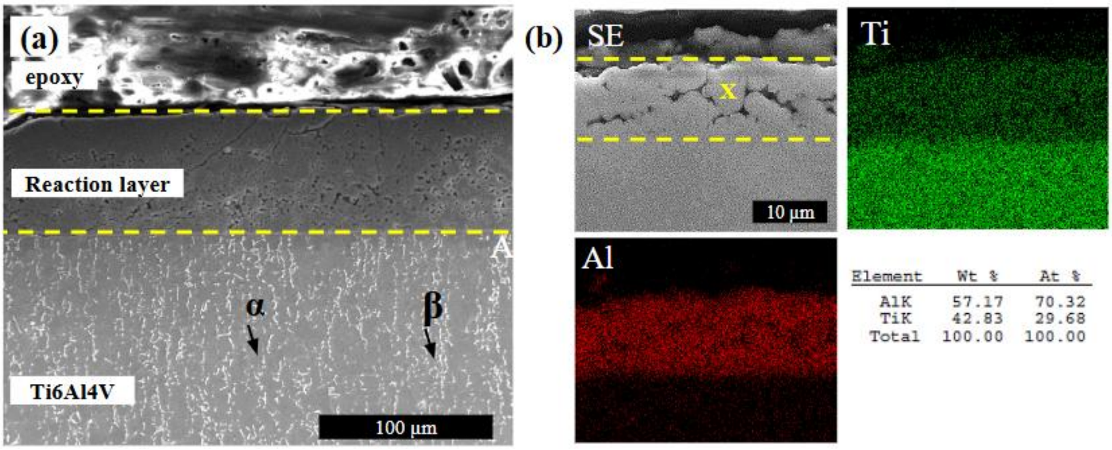


Figure 6. The change in the diameter and the contact angle of liquid AZ91 alloy at 800°C.

The SEM image of the reaction layer that developed after the wetting experiment, in which the liquid AZ91 alloy came into contact with the titanium alloy substrate, is given in Figure 7(a). Ti6Al4V alloy exhibits a V-rich β-phase (bright areas) and an Al-rich α-phase (dark areas) in the microstructure. In contrast to magnesium, a distinctive reaction layer was seen on the surface of the Ti-alloy, which had a thickness of about 70 µm. Additionally, EDS map analysis, Figure 7(b), revealed that this porous reaction layer was richer in titanium and aluminum. Moreover, Ti- and Al-enriched phases are thought to have formed as a result of aluminum from the

AZ91 alloy diffusing into the Ti6Al4V substrate at elevated temperatures. The presence of a $TiAl_3$ phase was identified as a likely product of this reaction, which was also confirmed by XRD analysis, Figure 4(b).



Element	Wt %	At %
AlK	57.17	70.32
TiK	42.83	29.68
Total	100.00	100.00

Figure 7. (a) SEM and (b) EDS spot analysis shown by X, and EDS map analysis of the reaction layer.

4. Conclusions

Based on the wetting test result, liquid magnesium and AZ91 alloy have favorable wetting behavior on the surface of Ti6Al4V alloy. Both magnesium and the AZ91 alloy have observed equilibrium contact angles of about 12°. Following liquid magnesium wetting on the Ti6Al4V substrate, TiO_2 reduction by liquid Mg caused MgO formation in addition to the Ti_3O phase at the interface. The $TiAl_3$ phase, along with the TiO and MgO phases, was formed as a result of the AZ91 alloy diffusing into the titanium substrate. These results demonstrate that liquid magnesium and its alloy can effectively infiltrate the Ti6Al4V structure, highlighting its potential for use in the fabrication of Ti-Mg composites.

Acknowledgements

Author would like to special thank to Ph.D. thesis co-supervisor.

Declaration of Competing Interest

No conflict of interest was declared by the authors.

Authorship Contribution Statement

Ezgi Bütev Öcal: Writing, Methodology, Data Preparation, and Editing.
Arcan F. Dericioglu: Methodology, Review and Editing, Supervision.

References

[1] A. W. Adamson and A. P. Gast, Physical Chemistry of Surfaces, 6th ed. New York, NY, USA: Wiley, 1997.
[2] E. Bütev-Öcal, “Designing of Ti-Mg composites for various applications,” Ph.D. dissertation, Dept. Metall. and Mater. Eng., Middle East Tech. Univ., Ankara, Turkey, 2020.
[3] J. Yang et al., “Material Processing and Design of Biodegradable Metal Matrix Composites for Biomedical Applications,” Ann. Biomed. Eng., 46, pp. 1229–1240, Jun. 2018, doi:10.1007/s10439-018-2058-y.
[4] M. Balog et al., “Titanium-Magnesium Composite for Dental Implants (BIACOM),” in TMS 2017 146th Annu. Meet. Exhib. Suppl. Proc., pp. 105–116, Feb. 2017, doi:10.1007/978-3-319-51493-2.
[5] G. Jiang et al., “Porous titanium with entangled structure filled with biodegradable magnesium for potential

biomedical applications," Mater. Sci. Eng. C., vol. 47, pp. 142–149, Feb. 2015, doi:10.1016/j.msec.2014.11.014.

[6] Z. Esen et al., "Corrosion behaviors of Ti6Al4V-Mg/Mg-Alloy composites," Corros. Sci., vol.166, 108470, Apr.2020, doi: 10.1016/j.corsci.2020.108470.

[7] Standard Specification for Titanium and Titanium-6 Aluminum-4 Vanadium Alloy Powders for Coatings of Surgical Implants, ASTM Standard F1580-01, 2006.

[8] N. V. Ravi Kumar et al., "Effect of alloying elements on the ignition resistance of magnesium alloys," Scr. Mater., vol. 49, pp. 225–230, Aug. 2003, doi:10.1016/S1359-6462(03)00263-X.

[9] K. Kondoh et al., "Wettability of pure Ti by molten pure Mg droplets," Acta Mater., vol. 58, pp. 606–614, Jan.2010, doi:10.1016/j.actamat.2009.09.039.

[10] D. Muscat and R.A.L. Drew, "Modeling the infiltration kinetics of molten aluminum into porous titanium carbide," Metall. Mater. Trans. A., vol. 25, pp. 2357–2370, Nov.1994, doi:10.1007/BF02648856.

[11] S. Bao et al., "Wetting of pure aluminium on graphite, SiC and Al_2O_3 in aluminium filtration," Trans. Nonferrous Met. Soc. China, vol. 22, pp. 1930–1938, Aug.2012, doi:10.1016/S1003-6326(11)61410-6.

[12] N. Eustathopoulos, "Wetting by Liquid Metals - Application in Materials Processing: The Contribution of the Grenoble Group," Metals, vol. 5, pp. 350–370, Mar. 2015, doi:10.3390/met5010350.

[13] Q. Tan et al., "Oxidation of magnesium alloys at elevated temperatures in air: A review," Corros. Sci., vol. 112, pp. 734–759, Jun. 2016, doi:10.1016/j.corsci.2016.06.018.