Mechanical Properties Enhancement of AZ91 Magnesium Alloy Reinforced with Various Ratios of Titanium Particles and Processed by ECAP

Song-Jeng Huang*, Yu-Quan Lee and Yopi Yusuf Tanoto

Department of Mechanical Engineering, National Taiwan University of Science and Technology, Taipei 10607, Taiwan

Abstract: This study utilized AZ91 magnesium-aluminum alloy as the matrix for magnesium-based composites, reinforced with micron-sized titanium (Ti). Gravity casting and mechanical stirring were employed to fabricate specimens with 0 wt.%, 0.3 wt.%, and 0.5 wt.% Ti reinforcement. Heat-treated samples underwent Equal Channel Angular Pressing (ECAP), and microstructures were analyzed via SEM and XRD. The experimental results demonstrate that the addition of micron-sized titanium improves the yield strength, ultimate tensile strength, and hardness. The inclusion of 0.5 wt.% titanium powder resulted in a 17.5% increase in ultimate tensile strength and a 37% increase in yield strength. After secondary processing with ECAP, the ultimate tensile strength showed an additional 25% increase, while the yield strength increased by 13.5%. Vickers hardness test results reveal a significant 13.7% strength improvement with the addition of 0.5 wt.% titanium powder, and after ECAP secondary processing, there was a marginal additional increase of 0.8%.

Keywords: Metal matrix composite, AZ91, titanium, mechanical properties, ECAP.

1. INTRODUCTION

Magnesium alloys and titanium alloys are widely used lightweight materials in industries such as aerospace, automotive, and healthcare. Magnesium alloys are known for their low density, strong mechanical properties and secondary process [1-3] though they have limited corrosion resistance [4]. While titanium alloys exhibit excellent corrosion resistance and high-temperature stability but are relatively heavy [5]. Metal matrix composites, combined lightweight properties with corrosion resistance [6], find extensive applications across various sectors [7]. Many researchers aim to reduce costs, rapidly enhance industrial capabilities, and integrate magnesium alloy composites into daily life, supporting the development of modern society.

Magnesium alloys, renowned for their lightweight properties and robust mechanical characteristics [8, 9], have been subject to extensive research to further enhance their performance. The addition of various reinforcing elements has been a focal point in this pursuit. Studies by Abbas A and Huang SJ explored the effects of Equal Channel Angular Pressing on the microstructure and mechanical behavior of annealed WS_2/AZ91 metal matrix composites [10]. Additionally, Pu D. *et al.* investigated the impact of Ti particles on the microstructure and mechanical properties of Ti/AZ91 composites, contributing valuable insights to the field [11].

Understanding the influence of interfacial structure on grain refinement and strength is crucial for optimizing magnesium alloy composites. The work by Ye J. *et al.* delves into the effect of interfacial structure on grain refinement and strength in Ti particle-reinforced AZ31 composites [12]. Furthermore, the study by Yuan Q hong *et al.* explored the interfacial structure in AZ91 alloy composites reinforced by graphene nanosheets, shedding light on innovative reinforcement strategies [13]. In the quest for enhanced microstructures and mechanical properties, Cui J. *et al.* investigated the optimization of AZ81 alloy by adding TC4 particles, providing valuable insights into the improvement of magnesium alloy performance [14].

Despite the significant strides in magnesium alloy research, a notable gap exists as no study to date has concurrently utilized titanium (Ti) powder and ECAP in the fabrication of AZ91 composite materials. This unexplored avenue presents a promising frontier for future research, offering the potential for groundbreaking advancements in the field of magnesium alloy composites. The absence of such dual methodologies in existing literature underscores the untapped potential and the need for further exploration in enhancing the mechanical properties of AZ91 through the synergistic combination of Ti powder reinforcement and ECAP processing.

*Address correspondence to this author at the Department of Mechanical Engineering, National Taiwan University of Science and Technology, Taipei 10607, Taiwan; E-mail: sgjghuang@mail.ntust.edu.tw

Table 1: Nominal Chemical Composition of the Investigated AZ91 Alloy [7]

Elements	Al	Zn	Mn	Fe	Si	Cu	Ni	Mg
Wt. % Conentrayion	8.95	0.84	0.26	0.005	0.009	0.0008	0.0007	Balance

2. EXPERIMENTAL

2.1. Material

Titanium (Ti) powder is produced by ATOMIC CRAFT with a particle size of 106-180μm. The composition of magnesium alloy AZ91 is listed in Table **1**. Meanwhile, AZ91 alloy was purchased from Jiehan Technology Corporation, Taichung, Taiwan [7].

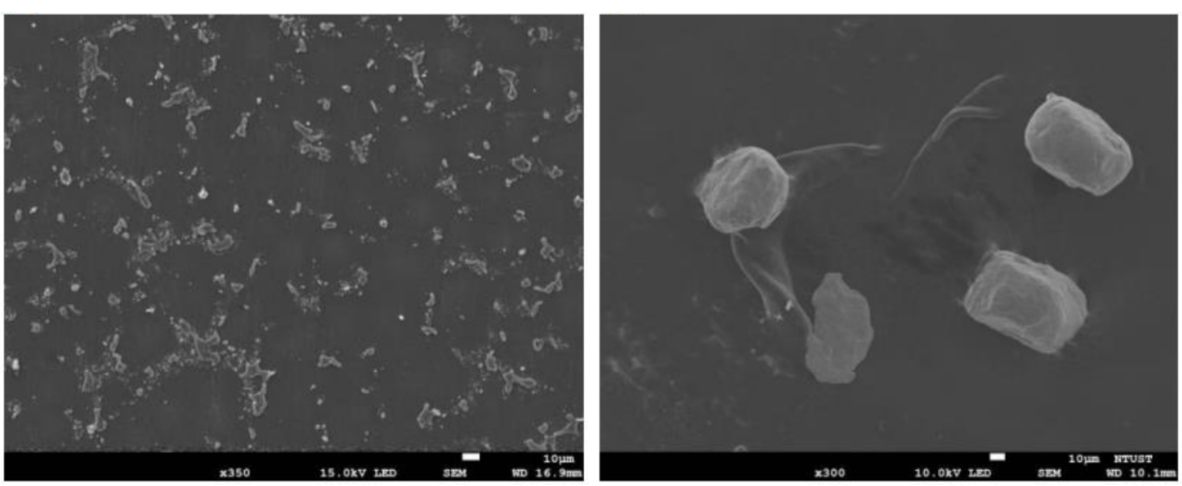
2.2. Composite Preparation

Titanium (Ti) was incorporated into the AZ91 magnesium alloy matrix as a reinforcing element at different ratios of 0 wt.%, 0.3 wt.%, and 0.5 wt.%. Ingot samples were produced through a combination of gravity casting and mechanical stirring methods. The melting process occurred in a resistance melting furnace, with both the matrix and reinforcing materials placed inside. The temperature was raised to 400°C, and SF6 gas was introduced, followed by the introduction of argon gas at 600°C to protect the molten metal. The mixture was stirred for 20 minutes at 750°C to disperse the reinforcing material. After a 10-minute rest, the molten material was poured into molds, left to naturally cool, completing the casting process.

To undergo Equal Channel Angular Pressing (ECAP), a T4 heat treatment was conducted. The heat treatment process involved gradually heating the samples at a rate of 5°C/min to 260°C, maintaining this temperature for 1 hour to release residual stresses. Subsequently, a slow heating process at a rate of 1°C/min to 400°C took approximately two hours, followed by a 10-hour dwell at this temperature. After heat treatment, the samples were cut into ECAP test bars. The ECAP process utilized Bc route and a 120° die. Initially, the test bar was preheated in a 120° die at 350°C for 40 minutes. After preheating, extrusion was performed, and the bar was water-quenched. According to Bc route, this process was repeated three times, completing the ECAP with 4 passes of extrusion.

2.3. Characterization

A field emission scanning electron microscope (FE-SEM), model JSM-7900 from Jeol Ltd., Japan, was utilized to investigate the morphology of AZ91 and Ti powders. The X-ray diffraction (XRD) patterns of both the original materials and the composites were examined using the D2 Phaser instrument from Bruker. The scanning range (2θ) for AZ91 powder was set from 25° to 75° with a step size of 0.05° and a scanning rate of 10°/min. Tensile testing was conducted using the Material Test System MTS-810 universal tensile testing machine from National Instruments Corporation. Surface properties were observed using a microhardness tester, Wilson HV1102, with an applied pressure of HV 0.1 kgf for a 10-second indentation period.



(**a**) (**b**)

Figure 1: SEM images of (**a**) Pure AZ91 and (**b**) Ti particle as received.

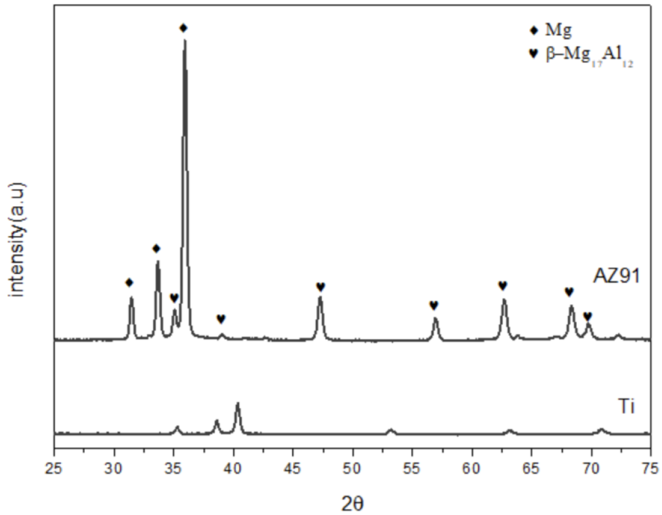


Figure 2: XRD pattern of AZ91 and Ti.

3. RESULT AND DISCUSSION

3.1. Material Characterization

The following images (Figure **1a**) are scanning electron microscope (SEM) images of magnesium alloy AZ91. The small particles observed in the image represent the second phase Mg17Al12 in the magnesium alloy. Excessive amount of the second phase causes an increase in hardness, a decrease in tensile strength, and a decrease in elongation of AZ91. The second image (b) is a scanning electron microscope (SEM) image of titanium powder. The addition of titanium powder to AZ91 not only reduces the grain size but also reduces the amount of the second phase.

XRD patterns of matrix, reinforcement, and composite materials are illustrated in Figure **2**. The AZ91 alloy exhibits prominent peaks corresponding to Mg phases and some $Al_{12}Mg_{17}$ phases. Titanium, serving as the reinforcement, demonstrates three major peaks. Figure **3** illustrates the XRD peak patterns of the composite materials. It is noteworthy that with an increase in the quantity of titanium powder, there is an observable enhancement in the peaks associated with titanium. In comparison to the XRD spectrum of the as-cast material, a reduction in the presence of the second phase is evident. However, there is not a significant alteration in the peaks related to magnesium.

3.2. Tensile and Hardness Test

Compared to AZ91, the UTS of composite materials is higher when an appropriate amount of reinforcing

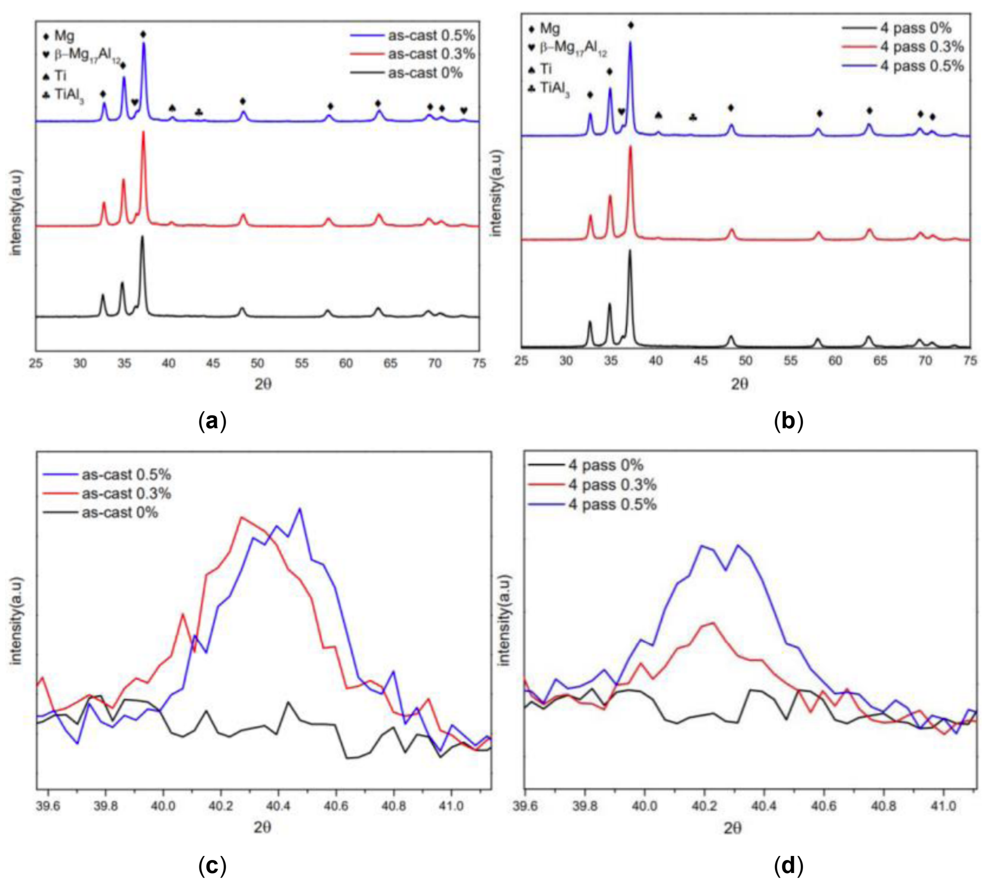


Figure 3: XRD pattern of (**a**) as-cast AZ91-Ti (**b**) ECAP 4pass AZ91-Ti (**c**) (**d**) peak of Ti.

material is added. Adding titanium (Ti) in weight percentages results in a gradual increase in UTS. The UTS of pure AZ91 is 177.4 MPa. However, after adding 0.3 and 0.5 weight percentages of Ti to AZ91, the UTS increases to 200.4 MPa and 208.5 MPa, respectively. After undergoing 4 passes of Equal Channel Angular Pressing (ECAP), the UTS further increases to 233.3 MPa, 244.5 MPa, and 260.8 MPa, respectively.

The observed increase in UTS with higher reinforcing material content demonstrates that Ti can enhance the mechanical properties of AZ91. After ECAP, a significant improvement in yield tensile strength was evident, with individual increases of 38.6%, 24.6%, and 13.5%. This is because ECAP can refine the grain size, and smaller grain sizes lead to higher yield strength [8][15][16].

Both yield strength (YS) and UTS exhibit similar trends. The yield strength of pure AZ91 is 114.4 MPa, whereas the addition of 0.3 wt.% and 0.5 wt.% of reinforcement material to the composite materials correspondingly increases the yield strength to 132.7 MPa and 156.7 MPa. After undergoing 4 passes of Equal Channel Angular Pressing (ECAP), the YS of pure AZ91 increased to 158.6 MPa, while the YS of composite materials with 0.3% and 0.5% additives increased to 165.4 MPa and 177.8 MPa, subsequently. The tensile strength of AZ91-Ti composite materials at different weight percentages is shown in Figure **4**.

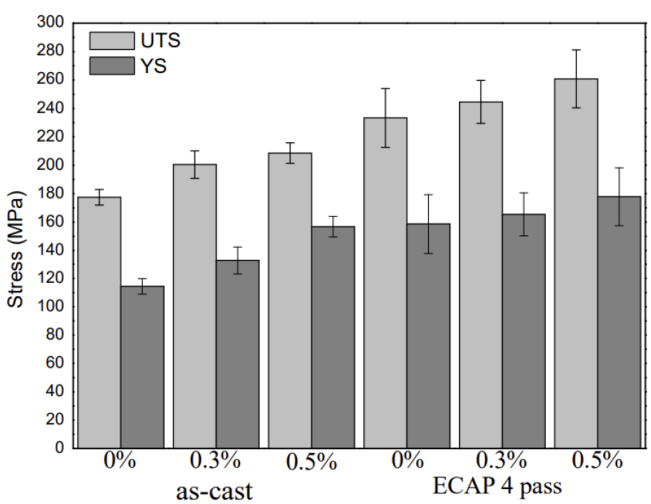


Figure 4: Yield and Ultimate tensile strength for AZ91-Ti composite.

Figure **5** shows the microhardness of AZ91 and AZ91-Ti composite materials at different weight percentages. It is worth noting that, compared to the T4 heat-treated condition, both the as-cast and post-ECAP composite materials exhibit higher microhardness values [13]. Furthermore, microhardness gradually increases with an increasing proportion of Ti reinforcing material. Specifically, the as-cast AZ91 has a microhardness of 74.36 HV, while the addition of 0.3 wt.% and 0.5 wt.% of Ti results in significant increases to 81.66 HV and 84.54 HV, respectively. After heat treatment, the microhardness decreases due to the dissolution of the second phase $Mg_{17}Al_{12}$ into the AZ91 substrate, resulting in values of 70.26 HV, 75.36 HV, and 78.94 HV. In the case of AZ91-Ti composite materials after ECAP, there is an improvement in microhardness. Interestingly, the hardness values are similar to the as-cast condition, measuring 71.58 HV, 80.06 HV, and 85.24 HV, subsequently.

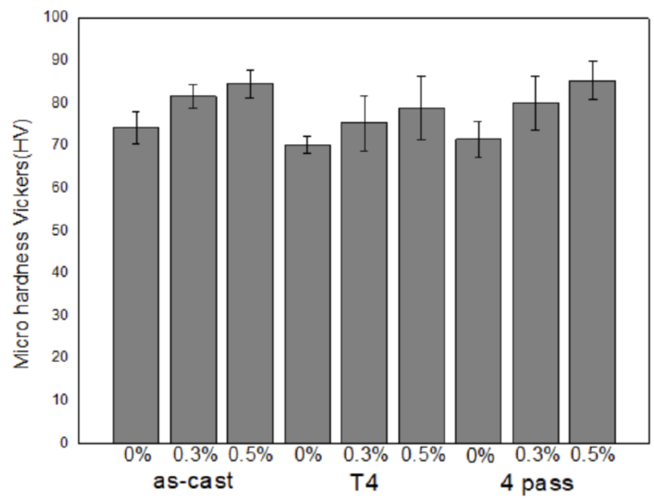


Figure 5: Micro hardness Vickers value for AZ91+Ti composite.

4. CONCLUSION

The experimental results revealed that the addition of micron-sized titanium as the reinforcing phase resulted in grain refinement, leading to improvements in yield strength, ultimate tensile strength, ductility, and hardness. In terms of yield strength, specimens without added titanium powder exhibited a yield strength of 114.4 MPa, while the addition of 0.3 wt.% and 0.5 wt.% titanium powder increased the yield strength by 16% and 37%, respectively. After ECAP 4 passes, compared to the as-cast condition, the yield strength increased by 38.6%, 24.6%, and 13.5% for the three respective compositions. Regarding ultimate tensile strength (UTS), specimens without added titanium powder had a UTS of 177.4 MPa. The addition of 0.3% and 0.5 wt.% titanium powder increased the UTS by 13% and 17.5%, respectively. After ECAP 4 passes, compared to the as-cast condition, the UTS increased by 31.5%, 22%, and 25% for the three respective compositions. Vickers hardness test results showed a significant improvement in hardness after adding 0.3% and 0.5% titanium powder, with increases of 11% and 13.7%, respectively. These findings contribute valuable insights into the microstructural evolution and enhanced mechanical properties of AZ91/Ti magnesium-based composite materials, shedding light on the potential applications of such composites in various engineering fields.

DECLARATION OF INTEREST STATEMENT

The authors declare that there isn't any conflict of interest with regard to the current study.

ACKNOWLEDGEMENT

The authors would like to express their gratitude to the National Science and Technology Council in Taiwan for their financial assistance provided through project grant number NSTC 111-2221-E-011-096-MY3.

REFERENCES

[1] Liu B, Yang J, Zhang X, Yang Q, Zhang J, Li X. Development and application of magnesium alloy parts for automotive OEMs: A review. Journal of Magnesium and Alloys 2023; 11: 15-47. https://doi.org/10.1016/J.JMA.2022.12.015

[2] Sehgal AK, Juneja C, Singh J, Kalsi S. Comparative analysis and review of materials properties used in aerospace Industries: An overview. Mater Today Proc 2022; 48: 1609-13. https://doi.org/10.1016/J.MATPR.2021.09.498

[3] Al-Zubaydi ASJ, Zhilyaev AP, Wang SC, Reed PAS. Superplastic behaviour of AZ91 magnesium alloy processed by high-pressure torsion. Materials Science and Engineering: A 2015; 637: 1-11. https://doi.org/10.1016/J.MSEA.2015.04.004

[4] Yun Y, Xue D, Halsall B, Vanooij W, Schulz M, Shanov V. The Effects of Primary Oxy-Salts on Anodizing Magnesium Alloy AZ91D. Journal of Coating Science and Technology 2015; 1: 78-87. https://doi.org/10.6000/2369-3355.2014.01.01.9

[5] Candan S, Unal M, Koc E, Turen Y, Candan E. Effects of titanium addition on mechanical and corrosion behaviours of AZ91 magnesium alloy. J Alloys Compd 2011; 509: 1958-63. https://doi.org/10.1016/J.JALLCOM.2010.10.100

[6] Huang SJ, Kannaiyan S. Carbon Nanotubes-Reinforced Magnesium Metal-Matrix Composites. Advances in Corrosion Control of Magnesium and Its Alloys: Metal Matrix Composites and Protective Coatings 2023: 75-89. https://doi.org/10.1201/9781003319856-8/carbon-nanotubes-reinforced-magnesium-metal-matrix-composites-song-JENG-huang-sathiyalingam-kannaiyan

[7] Huang SJ, Diwan Midyeen S, Subramani M, Chiang CC. Microstructure Evaluation, Quantitative Phase Analysis, Strengthening Mechanism and Influence of Hybrid Reinforcements (β-SiCp, Bi and Sb) on the Collective Mechanical Properties of the AZ91 Magnesium Matrix. Metals 2021; 11: 898. https://doi.org/10.3390/MET11060898

[8] Huang SJ, Wang CF, Subramani M, Fan FY. Effect of ECAP on Microstructure, Mechanical Properties, Corrosion Behavior, and Biocompatibility of Mg-Ca Alloy Composite. Journal of Composites Science 2023; 7: 292. https://doi.org/10.3390/JCS7070292

[9] Wang X, Wang X, Hu X, Wu K. Effects of hot extrusion on microstructure and mechanical properties of Mg matrix composite reinforced with deformable TC4 particles. Journal of Magnesium and Alloys 2020; 8: 421-30. https://doi.org/10.1016/J.JMA.2019.05.015

[10] Abbas A, Huang SJ. ECAP effects on microstructure and mechanical behavior of annealed WS2/AZ91 metal matrix composite. J Alloys Compd 2020; 835: 155466. https://doi.org/10.1016/J.JALLCOM.2020.155466

[11] Pu D, Wu S, Yang H, Chen X, Li J, Feng X, *et al.* Effect of Ti particles on microstructure and mechanical properties of TiP/AZ91 composites. Journal of Materials Research and Technology 2023; 22: 1362-74. https://doi.org/10.1016/J.JMRT.2022.12.028

[12] Ye J, Chen X, Luo H, Li J, Tan J, Yang H, *et al.* Effect of interfacial structure on grain refinement and strength of Ti particles reinforced AZ31 composites. Vacuum 2022; 203: 111287. https://doi.org/10.1016/J.VACUUM.2022.111287

[13] Yuan Q hong, Zhou G hua, Liao L, Liu Y, Luo L. Interfacial structure in AZ91 alloy composites reinforced by graphene nanosheets. Carbon N Y 2018; 127: 177-86. https://doi.org/10.1016/J.CARBON.2017.10.090

[14] Cui J, Yang H, Zhou Y, Tan J, Chen X, Song J, *et al.* Optimizing the microstructures and enhancing the mechanical properties of AZ81 alloy by adding TC4 particles. Materials Science and Engineering: A 2023; 863: 144518. https://doi.org/10.1016/J.MSEA.2022.144518

[15] Qiao XG, Ying T, Zheng MY, Wei ED, Wu K, Hu XS, *et al.* Microstructure evolution and mechanical properties of nano-SiCp/AZ91 composite processed by extrusion and equal channel angular pressing (ECAP). Mater Charact 2016; 121: 222-30. https://doi.org/10.1016/J.MATCHAR.2016.10.003

[16] Valiev RZ, Islamgaliev RK, Alexandrov IV. Bulk nanostructured materials from severe plastic deformation. Prog Mater Sci 2000; 45: 103-89. https://doi.org/10.1016/S0079-6425(99)00007-9

Received on 22-09-2023 Accepted on 21-10-2023 Published on 30-11-2023

https://doi.org/10.6000/1929-5995.2023.12.19