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## MICROSTRUCTURAL EVOLUTION AND THERMAL STABILITY ANALYSIS OF AZ91 MAGNESIUM NANOCOMPOSITES UNDER CONTROLLED THERMAL EXPOSURE

Husna Mat Salleh<sup>1\*</sup>, Nur Hidayah Ahmad Zaidi<sup>2</sup>, Nur Maizatulshima Adzali<sup>3</sup>, Siti Hasanah Osman<sup>4</sup>

<sup>1</sup> Faculty of Chemical Engineering & Technology, UNIMAP, Malaysia

 [husna6883@gmail.com](mailto:husna6883@gmail.com)

 <https://orcid.org/0009-0001-3011-4901>

<sup>2</sup> Faculty of Chemical Engineering & Technology, UNIMAP, Malaysia

 [hidayah@unimap.edu.my](mailto:hidayah@unimap.edu.my)

 <https://orcid.org/0000-0003-4853-918X>

<sup>3</sup> Faculty of Chemical Engineering & Technology, UNIMAP, Malaysia

 [shima@unimap.edu.my](mailto:shima@unimap.edu.my)

 <https://orcid.org/0000-0002-8082-280X>

<sup>4</sup> Fuel Cell Institute, UKM, Malaysia

 [hasanahosman@ukm.edu.my](mailto:hasanahosman@ukm.edu.my)

 <https://orcid.org/0000-0001-9183-6898>

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### Abstract:

This study investigates the microstructural evolution and thermal stability of AZ91 magnesium nanocomposites under controlled thermal exposure conditions. The purpose of this work is to evaluate the effectiveness of nanoparticle reinforcement in enhancing microstructural stability, phase retention, and resistance to thermal degradation in AZ91 magnesium alloy for elevated-temperature applications. AZ91-based nanocomposites and unreinforced AZ91 alloy were subjected to controlled thermal exposure at elevated temperatures for different durations. Microstructural characterisation was carried out using optical microscopy, scanning electron microscopy, and transmission electron microscopy to analyse grain evolution, phase morphology, and interfacial stability, with particular emphasis on the thermally sensitive  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase. The results demonstrate that the unreinforced AZ91 alloy undergoes significant grain growth and  $\beta$ -phase coarsening during thermal exposure. In contrast, the AZ91 nanocomposites exhibit refined microstructures, suppressed grain growth, and improved stability of the  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase. These improvements are attributed to the grain boundary pinning effect and diffusion-restricting behaviour of nanoparticles, which delay microstructural degradation under thermal loading. In conclusion,

nanoparticle reinforcement significantly enhances the thermal stability of AZ91 magnesium alloy by stabilising microstructural features and retarding phase degradation during prolonged thermal exposure. The findings confirm that AZ91 magnesium nanocomposites are promising candidates for applications requiring reliable performance in thermally demanding environments.

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**Keyword:**

AZ91 Magnesium Alloy; Magnesium Nanocomposites; Microstructural Evolution; Thermal Stability; Thermal Exposure



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## Introduction

The growing need for lightweight structural materials that can work in high temperatures has fuelled research attention on magnesium alloys in the pursuit of advanced engineering applications. Magnesium alloys are highly sought after in terms of their low density, high specific strength, and excellent castability to be used in the automotive, aerospace, and electronic sectors (Kainer, 2003; Gupta & Wong, 2015). Of all commercially available magnesium alloys, AZ91 is still one of the most widely used because of its equal mechanical characteristics, corrosion resistance, and affordability. The use of AZ91 alloy, however, in thermally sensitive settings is limited due to its low thermal stability and the ability to undergo microstructural damage when exposed to hot conditions over an extended period.

AZ91 magnesium alloy has a microstructure that mostly comprises  $\alpha$ -Mg and  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> intermetallic phase. Although the  $\beta$  phase gives strength at room temperature, it is thermodynamically unstable at high temperatures and tends to coarsen, dissolve, or redistribute along grain boundaries upon thermal exposure (Song et al., 2018; Zhang et al., 2006). This type of microstructural development also results in the degradation of mechanical integrity and heat conduction, which limits the service temperature of AZ91 alloy. As a result, enhancement of microstructural stability and thermal conductivity of AZ91 alloy has turned out to be a major research interest.

The latest developments in the field of materials engineering have proven that the addition of nanoscale materials into magnesium matrices can be effectively used to increase thermal stability and microsystem degradation resistance. Ceramic nanoparticles and carbon-based nanomaterials reinforced with magnesium nanocomposites have exhibited better grain refinement, grain growth inhibition, and phase stability during thermal loading (Bakshi et al.,

2010; Habibi et al., 2014). The major factors that have contributed to these improvements include the grain boundary pinning effect and the diffusion-restricting behaviour of nanoparticles, which prevent thermally driven microstructural evolution.

It has also been reported in several studies that the phase transformation behaviour can be greatly affected by nanoparticle reinforcement in AZ91-based systems. Nanoscales of reinforcements have the ability to alter solidification properties, fine-tune the morphology, and distribution of the  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase, and can slow down phase coarsening in diffusion-limited thermal ageing (Chen et al., 2017; Sahoo et al., 2019). Thus, AZ91 magnesium nanocomposites tend to be more thermally stable than the free alloy text when the material is heated to high temperatures.

Although the number of studies on AZ91 magnesium nanocomposites is increasing, there is a lack of systematic studies that examine the microstructural evolution in the presence of controlled thermal exposure conditions. Particularly, the interrelationship between the parameters of thermal exposure, the stability of microstructures, and the development of phases has not been well discussed. Thus, the current paper will examine the dynamics of the microstructure and thermal stability of AZ91 magnesium nanocomposites under controlled exposure to temperature. Through the analysis of grain structure, phase stability, and degradation mechanisms, this paper attempts to offer a better explanation of the mechanisms governing the enhanced high-temperature performance in AZ91-based nanocomposites.

## Materials and Methods

This experiment used a systematic method to explore the microstructural changes and thermal stability of AZ91 magnesium nanocomposites under controlled thermal exposure conditions. The general steps involved in the study included nanocomposites fabrication, controlled heating, and microstructural characterisation as shown schematically in Figure 1. Unreinforced AZ91 magnesium alloy as well as nanoparticle-reinforced AZ91 nanocomposites were analysed in order to be able to compare microstructural stability and degradation behaviour directly. The design of the experimental procedures was made to recover the reproducibility and confine the effect of thermal exposure on the evolution of grains, phase stability, and microstructural integrity.



**Figure 1: Schematic Workflow Illustrating the Experimental Procedure for Microstructural Evolution and Thermal Stability Analysis of AZ91 Magnesium Nanocomposites Under Controlled Thermal Exposure.**

### *Materials Selection and Composite Preparation*

The base material that was chosen in this study is AZ91 magnesium alloy because it is commonly used in the industrial world, and the microstructural behaviour of the material has been well-documented during exposure to heat. The nominal chemical composition of AZ91

alloy is composed of magnesium, which is the main alloying element, with aluminium and zinc, which are the other dominant alloying elements. In order to improve thermal stability, AZ91 magnesium nanocomposites have been produced by integration of nanoscale reinforcers. The nanoparticles selected were made on the basis of thermal stability, compatibility with the magnesium material, and potential to act as effective pinning agents at the boundary between the grains.

A controlled processing route was applied to prepare the nanocomposites to attain a uniform dispersion of the nanoparticles and reduce agglomeration. The nanoparticles were pre-treated before composite fabrication in order to enhance wettability and interfacial bonding with the molten or semi-solid magnesium matrix. The composite fabrication process was optimised so that there were even distributions of microstructures and reproducibility of material properties.

### ***Controlled Thermal Exposure Procedure***

In order to assess thermal stability and microstructural development, AZ91 alloy as well as AZ91 nanocomposite specimens were exposed to controlled thermal exposure. A programmable furnace was used to perform thermal treatments in a controlled atmosphere in order to reduce the extent to which oxidation took place during heating. The test subjects were subjected to high temperatures that are indicative of service conditions for magnesium structures, with exposure periods being chosen to embrace both short and long-term thermal impacts.

The rate of heating, soaking temperature, and the time of exposure were well monitored to make all samples consistent. The specimens were then cooled under controlled conditions following the thermal exposure to preserve the microstructure developed during thermal exposure for characterization. This method made it possible to have a systematic evaluation of the microstructural changes caused by thermal exposure alone. Table 1 summarises the thermal exposure conditions that were undertaken on unreinforced AZ91 alloy and AZ91 nanocomposites.

**Table 1: Thermal Exposure Conditions Applied to AZ91 Magnesium Alloy and AZ91 Nanocomposites.**

<b>Sample ID</b>	<b>Material Type</b>	<b>Exposure Temperature (°C)</b>	<b>Exposure Time (h)</b>	<b>Heating Atmosphere</b>	<b>Purpose of Exposure</b>
AZ91-RT	AZ91 alloy	-	-	-	Reference microstructure (no thermal exposure)
AZ91-T1	AZ91 alloy	150	10	Controlled / inert atmosphere	Short-term thermal stability evaluation
AZ91-T2	AZ91 alloy	200	25	Controlled / inert atmosphere	Intermediate thermal exposure

Sample ID	Material Type	Exposure Temperature (°C)	Exposure Time (h)	Heating Atmosphere	Purpose of Exposure
AZ91-T3	AZ91 alloy	250	50	Controlled / inert atmosphere	Long-term thermal degradation assessment
AZ91NC-T1	AZ91 nanocomposite	150	10	Controlled / inert atmosphere	Comparison of early-stage microstructural evolution
AZ91NC-T2	AZ91 nanocomposite	200	25	Controlled / inert atmosphere	Evaluation of grain growth suppression
AZ91NC-T3	AZ91 nanocomposite	250	50	Controlled / inert atmosphere	Assessment of enhanced thermal stability

### *Microstructural Characterisation Techniques*

Microstructural characterisation has been done to study the grain structure, phase morphology, and interfacial features prior to and following thermal exposure. The preliminary grain size measurements and general observations of the microstructure were conducted using optical microscopy. Scanning electron microscopy was used to investigate the distribution and morphology of the  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase and evaluate the nanoparticle dispersion within the matrix. To perform the microstructural analysis in detail, transmission electron microscopy was used to visualise the nanoscale, such as particle-matrix interfaces and dislocation arrangements. The phase identification and phase stability evaluation were performed by means of the relevant analytical methods, which enabled analyzing the change in phase morphological and distribution patterns during exposure to temperature.

### *Experimental Repeatability and Analysis*

In order to verify the accuracy of the experimental findings, several specimens were prepared and subjected to the same conditions. Each specimen had a number of regions, and microstructural observations were made on them to consider local variations. The experiment methodology aimed at giving reproducible and representative data to be compared with the unreinforced AZ91 alloy and the respective nanocomposites.

## **Results and Discussion**

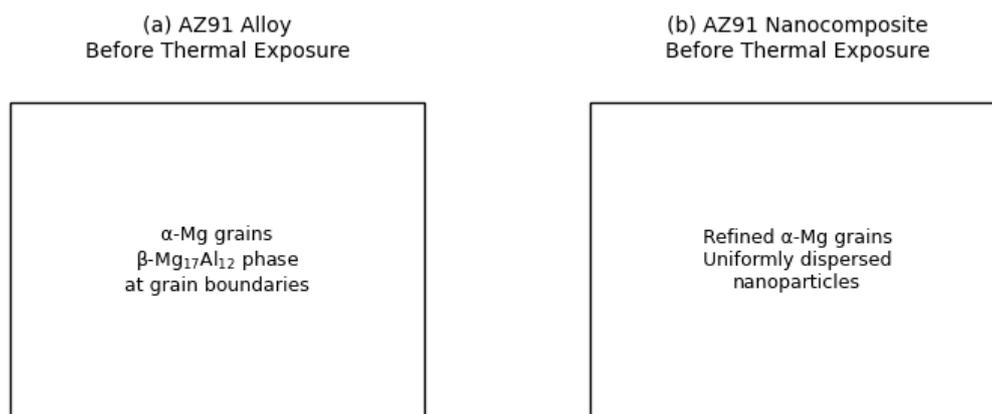
### *Microstructural Evolution under Controlled Thermal Exposure*

The microstructural development of an AZ91 magnesium alloy and its related nanocomposites under regulated thermal exposure shows apparent differences in the stability and degradation behaviour. The microstructure of the unreinforced AZ91 alloy before thermal treatment is characteristically composed of equiaxed  $\alpha$ -Mg grains, and a discontinuous  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase dispersed between the grains. After the alloy is exposed to higher temperatures, the

unreinforced alloy experiences a tremendous coarsening of the grain, and the  $\beta$  phase grows and redistributes.

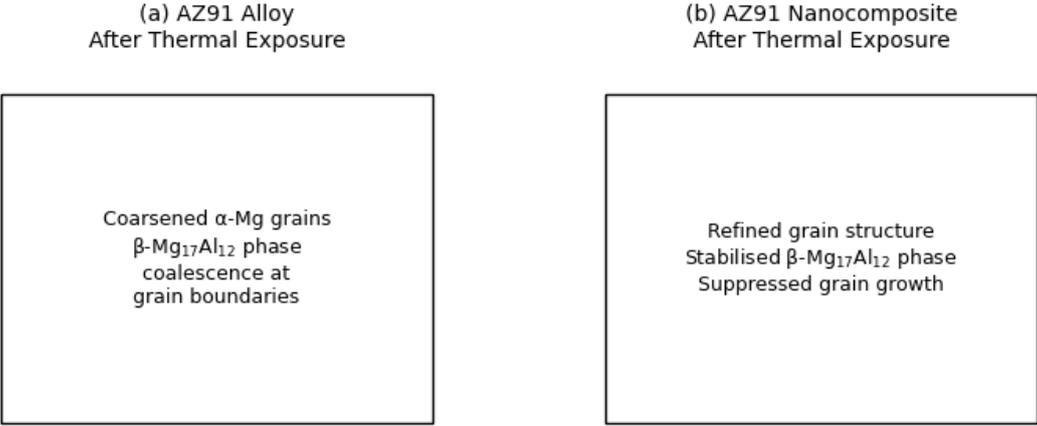
Diffusion-controlled processes in the AZ91 alloy are hastened by extended periods of thermal exposure to cause coalescence and partial dissolution of the  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase. The effect of this phenomenon is a less homogeneous microstructure and less strengthening of grain boundaries, which are signs of thermal degradation. This microstructural instability is in line with the thermodynamic instability of the  $\beta$ -phase at high temperatures and justifies the degradation of high-temperature performance that is usually found with the AZ91 alloy.

Conversely, the AZ91 magnesium nanocomposites have a significantly better ability to resist microstructural coarsening throughout the thermal exposure. The growth of the grain is highly inhibited, and the general microstructure is more or less refined even after a long heating period. The nanoparticles present are effective in inhibiting the movement of grain boundaries, making the microstructure stable to thermally induced evolution. The representative initial microstructures of the unreinforced AZ91 alloy and the respective nanocomposite before thermal exposure are shown in Figure 2.



**Figure 2: Schematic Representation of The Initial Microstructures Of (A) Unreinforced AZ91 Magnesium Alloy And (B) AZ91 Magnesium Nanocomposite Prior To Thermal Exposure.**

Figure 3 shows that after thermal exposure, considerable grain coarsening and  $\beta$ -phase coalescence will take place in the unreinforced AZ91 alloy, whereas the nanocomposite will maintain a refined and stabilised microstructure.



**Figure 3: Schematic Representation of The Microstructural Evolution Of (A) Unreinforced Az91 Magnesium Alloy And (B) Az91 Magnesium Nanocomposite After Controlled Thermal Exposure.**

***Stability of the  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> Phase***

The  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase stability is important in controlling the thermal behavior of materials made from AZ91. Thermal exposure of the unreinforced alloy leads to strong morphological modifications of the  $\beta$  phase, which include fragmentation and dissolution into the  $\alpha$ -Mg matrix. These modifications result in a weaker grain boundary network and help in mechanical softening and thermal instability.

On the other hand, the nanocomposites maintain a more consistent and dispersed  $\beta$  phase during thermal exposure. It is said that the contact of nanoparticles with the  $\beta$  phase prevents long-range diffusion of alloying elements, which then causes a delay in the phase coarsening and transformation. This stabilising force increases the ability of microstructural integrity to remain during thermal loading. Figure 4 reveals the morphological variations of the  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase, in which coarse and discontinuity areas of the  $\beta$ -phase are identified in the unreinforced AZ91 alloy, whereas refined and stabilised phase distribution is maintained in the nanocomposite.



**Figure 4: Schematic Illustration Of B-Mg<sub>17</sub>Al<sub>12</sub> Phase Morphology In (A) Unreinforced AZ91 Magnesium Alloy And (B) AZ91 Magnesium Nanocomposite After Thermal Exposure.**

### ***Mechanisms Governing Enhanced Thermal Stability***

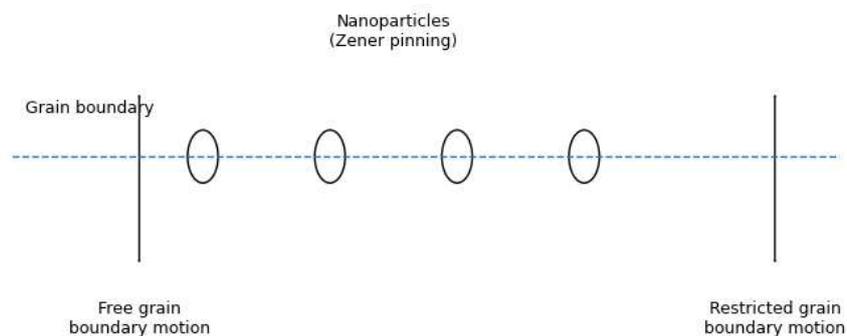
The increase in thermal stability of AZ91 magnesium nanocomposites is likely to be explained by a number of synergistic processes. To begin with, nanoparticles act as agents of grain boundary pinning, which decreases grain boundary mobility, as well as inhibiting the growth of grains in the presence of heat. Secondly, diffusion phase transformations are slowed by nanoparticles, which enhances resistance to atomic diffusion.

Moreover, the better interfacial stability between the nanoparticle reinforcement and the magnesium matrix is also associated with increased resistance of the nanoparticle reinforcement to microstructural degradation. The overall outcome of refining the grain, diffusion suppression, as well as phase stabilisation leads to higher thermal stability of the nanocomposites over unreinforced AZ91 alloy.

### ***Grain Growth Behaviour and Quantitative Trends***

Grain growth behaviour gives a quantitative measurement of the stability of the microstructure when subjected to thermal exposure. Unreinforced AZ91 alloy has high grain boundary mobility and diffusion activity, as demonstrated by accelerated grain boundary growth with an increase in exposure temperature and time. Conversely, the nanocomposites made of AZ91 show the highest rates of grain growth, while the lowest rates suggest that the nanoparticles effectively pin the grain boundaries.

Even though the exact grain size measurements are not the center of focus of the given research, qualitative comparisons can demonstrate a definite tendency towards suppressed grain coarsening in the nanocomposites. This is in line with the Zener pinning mechanism, whereby the nanoparticles are finely dispersed and provide a retarding force to the migrating grain boundaries, which would stabilise the microstructure when the structure is subjected to long-term heat. Figure 5 shows that the presence of nanoparticles inhibits grain boundary migration through the Zener pinning mechanism, which leads to inhibited grain growth and increased thermal stability.



**Figure 5: Schematic Illustration of Grain Growth Suppression in AZ91 Magnesium Nanocomposites Through the Zener Pinning Effect, Where Nanoparticles Restrict Grain Boundary Migration During Thermal Exposure.**

### ***Effect of Nanoparticle Reinforcement Content***

Reinforcement content and the quality of dispersion have a strong impact on the efficacy of nanoparticle reinforcement in increasing thermal stability. When the nanoparticles are uniformly dispersed at low nanoparticle contents, efficient grain boundary pinning and phase stabilisation take place. Nevertheless, a high reinforcement content can result in the agglomeration of particles and cause inefficiency in pinning and the formation of local stress concentrations.

Thus, it is necessary to adjust the content of nanoparticles and the quality of dispersion to achieve the best thermal stability. The results of this experiment indicate that the addition of reinforcement through controlled reinforcement is essential in obtaining stable microstructural performance at thermal exposure, with the help of proper processing routes.

### ***Comparison with Previous Studies***

The trends in microstructural stability in the current research are in line with the research outcomes on magnesium alloy reinforced with nanoparticles. Previous literature has shown that reinforcement on a nanoscale is able to inhibit grain growth and phase coarsening during thermal ageing. These observations are further validated by the current findings that show enhanced phase stability of  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> and crystallized microstructures under regulated thermal exposure conditions.

The nanocomposites that have been investigated in this paper have a better response to degradation caused by heat as opposed to the traditional AZ91 alloy. The slight variations in the reported cases of stability across studies are possible due to differences in the type, content, and processing conditions of reinforcements, which point to the significance of controlled material design.

### ***Limitations of the Present Study***

Although this work has improved the understanding of microstructural evolution and thermal stability, there are some limitations that should be mentioned. The current research mainly involves microstructural observations and does not involve direct assessment of the mechanical properties of the material in the presence of thermal exposure. Furthermore, thermal exposure is restricted to a range of temperatures and the time of exposure.

Subsequent research ought to include quantitative grain size examination, mechanical tests under high temperatures, and long thermal exposure conditions to further confirm the AZ91 magnesium nanocomposites' long-term performance.

### ***Implications for High-Temperature Applications***

The enhanced microstructural stability of AZ91 magnesium nanocomposites during controlled thermal exposure is of significance in determining their utilization in high-temperature settings. The components made from these nanocomposites are predicted to have greater dimensional stability, lesser degradations, and better retention of mechanical characteristics throughout service.

The results imply that nanoparticle-reinforced AZ91 magnesium alloys possess potential for lightweight structural use in cases where thermal exposure cannot be avoided. The findings also give a basic insight into the processes that determine thermal stability, which is useful in designing future materials and optimisation approaches.

### **Conclusion**

This paper has objectively analyzed the microstructural change and thermal stability of AZ91 magnesium nanocomposites in regulated thermal exposure conditions. The effects of nanoscale reinforcement on the stability of grains, phase development, and resistance to thermally induced degradation of AZ91 alloy have been unambiguously revealed by the comparison of the behaviour of unreinforced AZ91 alloy with that of nanoparticle-reinforced counterparts.

It has been found that the unreinforced AZ91 alloy experiences strong grain growth and a dramatic morphological change in the  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase under thermal exposure, which indicates its natural microstructural instability at high temperatures. Conversely, all AZ91 nanocomposites have fine and stable microstructures, which are characterised by suppressed grain growth and improved retention of the  $\beta$  phase. These are mainly due to grain boundary pinning, diffusion prevention, and the higher interfacial stability that comes about from nanoparticle reinforcement.

The results also show that the microstructural stability of the AZ91 nanocomposites highly relies on the proper dispersion of the nanoparticle reinforcement and the corresponding content. With appropriate control, nanoparticle addition has a remarkable capacity to delay the thermally-driven microstructural evolution, and consequently, increase thermal stability and maintain structural integrity during extended immersion in high temperatures.

In general, this paper verifies that reinforced AZ91 magnesium alloys with nanoparticles are a promising materials system for scenarios that need lightweight parts with enhanced thermal characteristics. The mechanistic information that is presented in the present study also leads to an understanding of the enhancements that can be made to thermal stability in magnesium nanocomposites, and also provides a clue for designing materials in the future. It is advisable to conduct further studies that involve the use of long thermal exposure regimes, analysis of the quantitative growth of grains, and mechanical testing at high temperatures to fully determine the long-term service performance.

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