Optimization of Micro-arc Oxidation Parameters for Preparing AlN Complex Ceramic Coatings on 1060 Al Substrate

Cuiling Fan¹,², Benkui Gong¹,*, Lei Wang¹, Shumin Xu², Xiaopeng Chen², Xiangkui Yang², Yaokun Pan¹, Rui Feng¹

1 School of Material Science and Engineering, Shandong University of Technology, Zibo, China.
2 Shandong Jinbao Electronics Co., LTD., Yantai, China

*Corresponding author e-mail: gongben69@163.com

Abstract. In this paper, AlN complex ceramic coatings are prepared on the surface of 1060 Al by micro-arc oxidation (MAO), selecting the current density, the AlN additive amount and NaOH concentration as parameters. In order to analyze the influence of three parameters on comprehensive performance of the ceramic coating, nine models are designed via a L₉(3³) orthogonal array. The surface morphology and phase composition of the MAO complex ceramic coatings are examined by scanning electron microscopy (SEM) and X-ray diffraction (XRD) respectively. Roughness, coating thickness and bonding strength of the Al substrate to the MAO complex ceramic coatings are tested and analyzed respectively. We present the results as follow: a) the current density has a relatively large influence on the roughness of the coatings, the minimum roughness of the coating is 0.034 μm. b) NaOH solution concentration has greater impact on the bonding strength of the coatings. The maximum critical load is 29.375 N. c) AlN additive amount has the largest impact on thickness and thermal conductivity of the coatings. The biggest thickness is 15.4 μm, and the maximum thermal conductivity is 3.9436 W/(mK). The corrosion resistance of the MAO samples is obviously improved.

1. Introduction

Micro-arc oxidation (MAO) is a new surface treatment technology for the in-situ growth of oxidized ceramic coatings on the surface of metals such as Al, Ti, Mg, etc. developed from anodic oxidation. It can effectively improve the physical and chemical properties of the alloy surface [1-3]. There are many similarities between MAO and anodic oxidation, but MAO is completely different from anodic oxidation in coating formation mechanism and process characteristics. MAO is not an improved anodic oxidation technology. MAO has many advantages, such as in-situ growth of ceramic coatings on the surface of the alloy, for that reason the coating properties like coating thickness, surface morphology, porosity and the size of surface micropores are controllable. Moreover, the process is simple, efficient, no sample morphology requirement, weak alkalinity of electrolyte and no pollution to the environment [4-7]. MAO shows the broad prospects for its application relative to traditional surface modification techniques such as anodizing and plating.
Al alloy is widely used in aerospace, electronic power, transportation, construction and other fields, due to its corrosion resistance, high specific strength, easy processing and forming, excellent thermal conductivity and electrical conductivity etc. advantages. However, the wear resistance and corrosion resistance of Al materials are still not satisfied, and the surface need to be treated [8]. Among a variety of surface modification technologies that improve the corrosion resistance of Al alloys, MAO is recognized as one of the most practical and promising technologies. MAO is also one of the most suitable surface modification techniques to grow an Al₂O₃ ceramic coating with enhanced surface mechanical properties on the Al surface. The performance of MAO ceramic coating mainly depends on electrolyte composition, electrical parameters, oxidation time and other factors. Many scholars have done a lot of research on these factors and have obtained some valuable results [9-13]. The MAO ceramic coating is porous, and the performance of the composite ceramic coating can be improved by adding appropriate amounts of TiO₂, SiO₂, SiC, ZnO, ZrO₂ etc., such as significantly reducing porosity, improving corrosion resistance and density of coatings, etc. [14]. That can effectively improve the physical and chemical properties of Al surface. AlN has attracted much attention for its high thermal conductivity, low dielectric constant, high electrical resistivity, thermal expansion coefficient similar to silicon, and excellent electromechanical properties. It is the most promising new generation of highly thermally conductive ceramic substrates currently [15]. In this work, the effect of AlN on the properties of the MAO complex ceramic coatings is investigated by adding different amounts of AlN powder into the electrolyte.

MAO is a complex electrochemical and plasma electrochemical process. The electrochemical parameters and electrolyte composition have a significant impact on it. For optimization of MAO process parameters, current density and the AlN additive amount and NaOH concentration in conventional silicate electrolyte system as MAO process parameters were designed by three factors and three levels of orthogonal test. Thence, optimal process parameters of MAO on Al surface were studied by orthogonal experimental design. Via analyzing the morphology, microstructure and composition of the complex ceramic coating, and the results of the roughness, adhesive strength, thickness, thermal conductivity of the coating. The effects of these three process parameters on the surface properties of complex ceramic coatings is discussed, and the optimal results of MAO process parameters are obtained.

2. Experimental

2.1. Composite Ceramic Coating Preparation

In this work, 1060 Al with dimensions of 100×100×1.42 mm was selected as the substrates to be coated by MAO. Polished 1060 Al steps by step with 400#, 600#, 1000# sandpaper, hereafter polishing until the surface is smooth without obvious scratches (roughness is 0.5 μm).

Prior to MAO treatment, substrate was degreased by acetone and alcohol followed by rinsing 10 minutes with an ultrasonic cleaning machine (KX-1620QT, 40kHz). Then it is dried by using hairdryer to ensure that the substrate surface is clean and free of water residue.

According to the designed orthogonal test scheme, MAO ceramic coating was prepared on 1060 Al substrate. Three-level and three-factor orthogonal test is designed as Table 1. MAO treatment of the substrates according to the scheme. The MAO complex ceramic coatings were formed on Al substrate by using MAO equipment (WHD-30) with stirring electrolytic solution by mechanical agitator (IKA rw28) for 20 minutes. The MAO experimental system for this work is given in Figure 1. The electrical parameters were set to the current density is 2-5 A/dm², the frequency is 350 Hz, and the positive and negative duty ratios are both 30%. Electrolytic solution is composed of Na₂SiO₃ (AR), NaOH (AR), Na₃PO₄ (AR), EDTA-2Na (AR), AlN powders. The AlN powders with an average particle diameter of 3 μm commercially available.
2.2. Coating Characterization

The samples subjected to MAO were washed with deionized water and alcohol. Rinsed for 10 minutes with an ultrasonic cleaning machine. Samples after drying was tested and analyzed. The phase composition of the complex ceramic coatings was investigated using X-ray diffraction (XRD, Cu-Kα, Rigaku D/max 2500 PC, over 2θ ranges from 20° to 90° at a scan rate of 2°/min). The scanning electron microscopy (SEM, KYKY-2800B) was used to observe morphology of the sample surface.

The surface roughness (Ra) of the ceramic coatings was measured with surface roughness tester (TR100). The adhesion of the MAO complex ceramic coatings to the substrate was measured by automatic scratch instrument for coating adhesion (WS-2005) with load of 50 N, loading rate of 100 N/min, scratch rate of 3 mm/min with treatment time of 2 minutes. The interlace between the complex ceramic coatings and the substrate was observed by metallographic microscope (Axio Scope A1, Germany). Five positions were randomly selected to measure the thickness of the ceramic coating, and the average value was used as the experimental ceramic coating thickness result. Thermal conductivity of the ceramic coatings were tested by heat conduction modulus testing instrument (DRL-III). The corrosion resistance of the samples before and after MAO was evaluated by potentiodynamic measurement.

Table 1. Orthogonal experiment Table for prepare composite AlN ceramic coating.

| Sample number | Factor | Current density / (A/dm²) | AlN / (g/L) | NaOH / (g/L) |
|---------------|--------|--------------------------|-------------|--------------|
| 1#            | 2      | 0                        | 1           |              |
| 2#            | 2      | 2                        | 1.5         |              |
| 3#            | 2      | 5                        | 2           |              |
| 4#            | 3      | 2                        | 2           |              |
| 5#            | 3      | 5                        | 1           |              |
| 6#            | 3      | 0                        | 1.5         |              |
| 7#            | 5      | 5                        | 1.5         |              |
| 8#            | 5      | 0                        | 2           |              |
| 9#            | 5      | 2                        | 1           |              |
polarization curve in NaCl solution (3.5 wt%). The potentiodynamic polarization curve was conducted on an electrochemical workstation (CHI660A, Shanghai). Afterwards calculate corrosion current and corrosion potential.

3. Result and Discussion

3.1. Phase Composition of MAO Complex Ceramic Coating

X-ray diffraction patterns of 6# sample which with the biggest coating thickness treated by MAO are given in Figure 2. It is shown that the diffraction peak corresponding to the alloy substrate Al is still strong, indicating that the X-rays are relatively easy to penetrate the complex ceramic coatings. Then the peak of the substrate is detected, which is related to the thin ceramic coatings and the porous surface[16]. MAO complex ceramic coating contains typical phases $\alpha$-$\text{Al}_2\text{O}_3$, $\gamma$-$\text{Al}_2\text{O}_3$, SiO$_2$ and a small amount of AlN. A small amount of SiO$_2$ phase is generated by electrolysis of Na$_2$SiO$_3$ in electrolyte under the action of electric field to generate Si$^{4+}$, which reacts with O$^{2-}$ to generate SiO$_2$ and adsorbs on the surface of ceramic coating. AlN phase indicates that the AlN powder in the electrolyte has been successfully deposited in the ceramic coatings, and the reaction of MAO in Al substrates generates in situ complex ceramic coatings.

![X-ray diffraction patterns of the complex ceramic coatings.](image)

Figure 2. X-ray diffraction patterns of the complex ceramic coatings.

3.2. Structure of Complex Ceramic Coating

Surface morphologies of MAO complex ceramic coatings formed on 1060 Al under current density of 5 A/dm$^2$ with different amount of AlN as shown in Figure 3. Figures 3(a), (b), (c) show samples 8#, 9#, 7# surface microstructure respectively. During the MAO reaction, the substrate Al reacts with O$^{2-}$ ions in the electrolyte to form a reaction product mainly composed of Al$_2$O$_3$. When the molten reaction product is discharged into the electrolyte, it will be condensed by the electrolyte to form micropores as shown in Figure 3(a). The microcrack showed on Figure 3(a) is formed under the action of thermal stress during MAO. AlN additives are combined with the molten Al$_2$O$_3$ in the discharge channel under the action of electric field, so that the volume of the melt increases, and the insufficiently sintered AlN additives would block the discharge channel. When the ceramic coating is generating, AlN additives are deposited in the complex ceramic coatings, which can block the micropores generated by the discharge, can increase the density of the coatings, can improve the quality of the coatings consequently. Therefore, the size and quantity of the micropores were reduced as showed in Figures 3(b) and 3(c).
Figure 3. SEM images of surface morphology of the MAO ceramic with the same current density. 
(a) no-additive MAO ceramic coating; (b) 2 g/L AlN additive MAO complex ceramic coatings; (c) 5 g/L AlN additive MAO complex ceramic coatings.

The chemical compositions of the depositions formed on the MAO ceramic coatings surfaces under current density of 5 A/dm² with different amount of AlN, as determined by EDS, are shown in Figures 4(b), 4(d) and 4(f). From which it can be concluded that, when AlN is not added to the electrolyte, the surface elements of the ceramic coating mainly consist of O, Al and Si, as showed in Figure 4(b). When AlN is added to the electrolyte, AlN is deposited in the ceramic coating. The element composition of the ceramic coating is O, Al, N and Si. As the amount of AlN in the electrolyte increases, the content of element N increases significantly, as showed in Figures 4(d) and 4(f). Based on XRD and EDS results, it can be concluded that when AlN is not added to the electrolyte, ceramic coatings composition is Al₂O₃ and SiO₂. After adding AlN, the main components of the coating are Al₂O₃, SiO₂ and AlN. With the increase of AlN amount, the AlN component in the coating increases accordingly.

Figure 4. SEM and EDS images of surface morphology of the MAO ceramic with the same current density.
(a), (d) SEM and EDS analysis sample of 8th MAO ceramic coating; (b), (e) SEM and EDS analysis of sample 9th MAO ceramic coating; (c), (f) SEM and EDS analysis of sample 7th MAO ceramic coating.
3.3. Roughness of Complex Ceramic Coating

Roughness of complex ceramic coatings formed on 1060 Al under orthogonal conditions are shown in Figure 5. The minimum roughness is 0.036 μm, maximum roughness is 0.498 μm. The surface roughness of the complex ceramic coatings is lower than Al substrate. Ceramic coatings made by MAO treatment are smoothing than the Al substrate, its roughness has reduced. The range in the orthogonal test results represents the maximum difference of three levels of each factor, and reflects the influencing degree of the factors on complex ceramic coating roughness. On average, it reflects the the influencing degree of the different levels of the same factor on the MAO coating roughness. The bigger average indicated the level of greater influencing degree. The results of Table 2 show that the influencing degree of each factor on complex ceramic coatings roughness is current density, NaOH solution concentration, AlN solution concentration successively. The surface roughness could be related to the coating thickness, large micropores and protrusions formed on the coating surface. As the current density increases, ceramic coatings thickness with large micropores and protrusions grew together. The increase in temperature of the MAO caused by the current density increase facilitates the flow of the molten material to make the surface of the ceramic coatings flat and smooth. However, if the current density is too high, the breakdown of the ceramic coatings will be intensified, and volcanic protrusions and ridges are easily formed. This ceramic coating of the morphology is easily contracted and cracked under the chilling of the electrolyte. On the other hand, during MAO, the voltage increases as the current density increases, the ceramic coatings were covered by new growing ceramic coatings in succession, the micropore diameter gradually becomes larger, and the number of holes becomes smaller. At the same time, the electric spark will become more intense during the reaction which made the coatings surface larger micropores and protrusions, resulting in a rough surface. When the ceramic coating is formed, the AlN additive is concentrated in the ceramic coating surface, blocking the ion channel generated by the discharge, thereby increasing the density and smoothness of the MAO complex ceramic coatings. It can be concluded from Table 2, complex ceramic coatings prepared under the parameters of current density as 2 A/dm², 5 g/L AlN, 1 g/L NaOH have the smallest roughness. Verifying the results of the orthogonal test, the roughness of the complex ceramic coatings prepared under the conditions is 0.034 μm, in compliance with the results of the orthogonal test.

![Figure 5](image_url)

**Figure 5.** Effect of orthogonal experimental conditions on roughness of the complex ceramic coatings.
Table 2. The factor mean and range of the complex coatings roughness.

| Factor  | Current density | AlN    | NaOH   |
|---------|-----------------|--------|--------|
| Mean 1  | 0.203           | 0.325  | 0.216  |
| Mean 2  | 0.326           | 0.359  | 0.397  |
| Mean 3  | 0.381           | 0.225  | 0.297  |
| Range   | 0.181           | 0.134  | 0.178  |

3.4. Adhesive Strength of Complex Ceramic Coating

The Figure 6 shows the scratch test for the complex ceramic coatings. The minimum critical load is 13.4375 N, the maximum critical load is 29.3750 N, the complex ceramic coating is well bonded to the substrate. The structure of the interface region of the metal substrate composite is very complicated. When the metal substrate and the reinforcement are combined at high temperature, different degrees of interfacial reaction will occur. The metal substrate will also have element segregation, diffusion, solid phase transformation and other phenomenon, during cooling, solidification and heat treatment. The structure and properties of the interface play a decisive role in the stress and stress distribution of metal substrate composites, as well as the load transfer and fracture processes[22]. The surface of the Al substrate undergoes a series of reactions such as chemical, electrochemical, and plasma oxidation during MAO to form an alumina ceramic coating in situ with excellent interfacial adhesive strength. During MAO reaction, high temperature and pressure are occurred in the micro-arc region instantaneously, and strong diffusion are occurred at the interface between the Al substrate and generated oxide coating. The oxide coating and Al substrate are staggered and chimeric. In addition, MAO directly oxidizes the Al substrate into crystalline Al₂O₃. So that there is no large void at the interface of ceramic coating and Al substrate, and the lattice of them also matches well. Therefore the ceramic coating and Al substrate have strong adhesive strength[23]. The results of Table 3 show that the degree of influence of each factor on the adhesive strength of the complex ceramic coatings is NaOH solution concentration, current density, AlN solution concentration successively. NaOH concentration has a great effect on the growth of complex ceramic coating. Too high concentration of NaOH will lead to too intense micro-arc discharge on the surface of the ceramic coating during MAO, which will affect the quality of the ceramic coating. The critical load value ruptured the ceramic coating will decrease with the increase of current density. It can be concluded from Table 3, complex ceramic coatings prepared under the parameters of current density as 2 A/dm², 5 g/L AlN, 2 g/L NaOH have the maximum adhesive strength. It can be seen from Figure 6 that the adhesive strength of the sample 3⁴ is the maximum value of the prepared sample, which is in accordance with the results of the orthogonal test.
3.5. Thickness of Complex Ceramic Coating

Thickness of the complex ceramic coatings is shown in Figure 7. The minimum thickness of the complex ceramic coatings is 5.7 μm, maximum thickness is 14.7 μm. The formation and morphology changes of the ceramic coating are directly related to the breakdown of the coating and the temperature of the micro-arc zone. When the current density is large, the energy density at the surface of the sample is increased. The high-energy plasma generated by the high current density easily breaks down the already formed ceramic coatings, and accelerates the reaction and migration speed of the materials inside and outside the ceramic coatings. Thus the coatings formation speed is accelerated\cite{24}. Complex ceramic coatings are prepared at a relatively low current density conditions, so the thickness of the ceramic is thin. The results in Table 4 show that the influencing degree of each factor on the adhesive strength of the complex ceramic coatings is AlN amount, current density, NaOH solution concentration successively. As can be seen from Figure 2, the AlN additive has been successfully deposited in the coatings during the MAO reactions to form the MAO complex ceramic coatings. Therefore, an appropriate amount of AlN additive can have a high film thickness. It can be concluded from Table 4, complex ceramic coatings prepared under the parameters of current density as 5 A/dm\(^2\), 5 g/L AlN, 1 g/L NaOH have the maximum thickness. Verifying the results of the orthogonal test, the thickness of the complex ceramic coatings prepared under the conditions is 15.4 μm, in compliance with the results of the orthogonal test.

| Factor               | Current density | AlN   | NaOH  |
|---------------------|-----------------|-------|-------|
| Mean 1              | 21.475          | 16.309| 16.217|
| Mean 2              | 16.033          | 17.121| 15.092|
| Mean 3              | 15.809          | 19.887| 22.099|
| Range               | 5.666           | 3.578 | 6.917 |
Figure 7. Effect of orthogonal experimental conditions on thickness of the complex ceramic coatings.

Table 4. The factor level and range of the complex ceramic coatings thickness.

| Factor     | Current density | AlN     | NaOH   |
|------------|-----------------|---------|--------|
| Mean 1     | 10.267          | 12.433  | 12.167 |
| Mean 2     | 11.267          | 8.967   | 11.133 |
| Mean 3     | 13.633          | 13.767  | 11.867 |
| Range      | 3.366           | 4.800   | 1.634  |

3.6. Thermal conductivity

Figure 8 shows thermal conductivity of the ceramic coatings made by MAO on the surface of 1060 Al. The minimum thermal conductivity of the complex ceramic coatings is 1.7089 W/(mK), maximum thermal conductivity is 3.8208 W/(mK). Table 5 shows that the influencing degree of each factor on the adhesive strength of the complex ceramic coatings is AlN amount, current density, NaOH solution concentration successively. As the current density increases, the number and diameter of the ceramic coating micropores prepared by MAO also increase. The surface of the complex ceramic coating has microcracks and micropores as showed in Figure 3, so that the ceramic coating would have a lower thermal conductivity. In addition, AlN additive which has good thermal conductivity and small thermal expansion coefficient is a good thermal shock resistant material. The complex ceramic coating contains AlN as can be seen from Figure 2. In the complex ceramic coating, heat is transferred along the AlN-Al₂O₃ composites with lower thermal resistance. Complex ceramic coatings with AlN have well thermal conductivity. In consequence, the coatings can be used as an excellent heat transfer medium. It can be concluded from Table 4, the MAO complex ceramic coatings prepared under the parameters of current density as 5 A/dm², 5 g/L AlN, 2 g/L NaOH have the maximum thermal conductivity. Verifying the results of the orthogonal test, the thermal conductivity of the complex ceramic coatings prepared under the conditions is 3.9436 W/(mK), in compliance with the results of the orthogonal test.
Figure 8. Effect of orthogonal experimental conditions on the thermal conductivity of the complex ceramic coatings.

Table 5. The factor mean and range of the complex ceramic coatings thermal conductivity.

| Factor  | Current density | AlN  | NaOH |
|---------|----------------|------|------|
| Mean 1  | 2.722          | 2.304| 2.482|
| Mean 2  | 2.202          | 2.481| 2.522|
| Mean 3  | 2.755          | 2.893| 2.675|
| Range   | 0.553          | 0.589| 0.193|

3.7. Potentiodynamic Polarization Curves

Corrosion potential (Ecorr) and the corrosion current density (Icorr) are commonly used to describe the corrosion resistance of specimens. In general, the self-corrosion potential reflects the degree of difficulty of corrosion of the alloy. The self-corrosion current density reflects the corrosion rate of metal. The higher the self-corrosion potential and the smaller the self-corrosion current density, the better the electrochemical corrosion resistance of metal\(^{[26]}\). The potentiodynamic polarization curves of the substrate and the samples treated by micro-arc oxidation in NaCl solution (3.5 wt\%) are depicted in Figure 9. The electrochemical parameters of potentiodynamic polarization are summarized in Table 6. Pure Al material has low corrosion resistance, and the ceramic coating is formed in situ by micro-arc oxidation treatment, which can effectively improve the corrosion resistance of the pure Al material.

It can be seen that the Ecorr of the samples treated by MAO are obviously higher than the Al substrate from Figure 9 and Table 6. The Icorr of the MAO samples relative to Al substrate increase an order of magnitude. In the electrochemical corrosion process, Cl\(^-\) does not completely penetrate the entire ceramic coatings, and the inner dense coating can block it, so that it cannot be quickly corroded to the substrate, thereby protecting the Al substrate. Micropores are present in the ceramic coatings, and the corrosive medium is etched through the holes to the surface of the Al substrate to reduce the adhesive strength of the coating to cause peeling. In addition, AlN additives can also effectively reduce the number and size of holes in the ceramic coating. The complex ceramic coatings prepared by the experiment has a certain adhesive strength with the substrate, which can effectively hinder the destruction of the Al substrate and the ceramic coatings by the corrosive medium, thereby improving the corrosion resistance of the samples subjected to the MAO treatment.
Figure 9 Potentiodynamic polarization curves of the substrate and the MAO samples in 3.5 wt% NaCl solution.

Table 6 Analysis result based on potentiodynamic polarization curves in Figure 9.

| Sample | 1  | 2  | 3  | 4  | 5  | 6  | 7  | 8  | 9  | Substrate |
|--------|----|----|----|----|----|----|----|----|----|-----------|
| $I_{corr}/(\times 10^{-6} \text{A/cm}^2)$ | 7.357 | 6.012 | 1.826 | 7.283 | 4.91 | 2.104 | 1.260 | 1.331 | 1.612 | 16.12 |
| $E_{corr}/\text{V}$ | -0.929 | -0.689 | -0.989 | -0.743 | -0.702 | -0.746 | -0.728 | -0.720 | -0.634 | -1.434 |

4. Conclusion

The complex ceramic coating on the surface of the Al has micropores. Adding AlN additives can effectively reduce the number and size of micropores. The phase composition of the coatings formed on Al substrate with AlN additive mainly consist of $\alpha$-$\text{Al}_2\text{O}_3$, $\gamma$-$\text{Al}_2\text{O}_3$, AlN and Al.

Three factors and three levels orthogonal test results show that current density has a relatively large influence on the roughness. The scheme for preparing the complex ceramic coatings to obtain the minimum roughness is 2 A/dm$^2$, 5 g/L AlN, 1 g/L NaOH. The smallest roughness is 0.034 μm. NaOH solution concentration has a greater influence on the adhesive strength of the complex ceramic coatings. Complex ceramic coatings prepared under the parameters of current density as 2 A/dm$^2$, 5 g/L AlN, 2 g/L NaOH have the maximum adhesive strength. The maximum critical load is 29.375 N. The amount of AlN has the largest influence on thickness of the ceramic coating. Complex ceramic coatings prepared under the parameters of current density as 5 A/dm$^2$, 5 g/L AlN, 1 g/L NaOH have the biggest thickness. The biggest thickness is 15.4 μm. The amount of AlN morely influence on thermal conductivity of the complex ceramic coatings. The scheme for preparing the complex ceramic coatings to obtain the maximum thermal conductivity is 5 A/dm$^2$, 5 g/L AlN, 1 g/L NaOH. The maximum thermal conductivity is 3.9436 W/(mK).

The $E_{corr}$ of the samples treated by MAO are obviously higher than the Al substrate. The $I_{corr}$ of the MAO samples relative to Al substrate increases an order of magnitude. The corrosion resistance of the samples treated by MAO is obviously improved.

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