International Journal of ELECTROCHEMICAL SCIENCE www.electrochemsci.org

# Effect of process parameters on microstructure and properties of AlCoCrFeNi-WC-WS<sub>2</sub> composite coating prepared by laser cladding

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Received: 5 October 2020 / Accepted: 16 December 2020 / Published: 31 December 2020

To further enhance the hardness, wear, and corrosion resistance of high-entropy alloy (HEA) coatings, an AlCoCrFeNi-WC-WS<sub>2</sub> composite coating was prepared on the surface of cast iron using laser cladding technology. X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS) analyses as well as microhardness, friction and wear, and electrochemical tests were performed to characterize and analyze the phase structure, composition, and forming quality of coating. The effects of process parameters such as laser power and scan rate on the microstructure and properties of coating were evaluated. With the increase in laser power, the amount of unmelted powder, cracks, and pores gradually decreases inside the coating, and the thickness and density increase. The coating is mainly composed of face-centered cubic (FCC) phase, carbide-reinforcing phase, and WS<sub>2</sub> self-lubricating phase. WC and its in situ synthesized chromium and iron carbide increased the hardness of coating by 3-4 times of the substrate, and the maximum hardness can reach 1187.2 HV. WS<sub>2</sub> exists as the self-lubricating phase in the coating, ensuring the formation and stability of lubricating film during friction and wear, helping the coating have favorable wear resistance. In addition, a compact passive film is formed by the corrosion-resistant elements Co, Cr, and Ni, enabling the coating to have great corrosion resistance in 3.5 wt% NaCl solution.

**Keywords:** Composite coating; carbide reinforcing phase; in-situ synthesis; solid lubricant; self-lubricating phase

# **1. INTRODUCTION**

High-entropy alloy (HEA) is a new type of metal [1]. HEA has quickly become one of the hot topics in material research because of its unique phase and organizational structures, excellent performance, and new alloy design concepts since it was proposed in 2004. HEA is mainly prepared in three forms: bulk metal, film, and coating. HEA films and coatings can not only achieve excellent

performance, but also reduce the use of materials and costs compared with block HEA. However, the effective thickness of HEA films does not satisfy the requirements of industrial production. In contrast, the study of HEA coating has the advantage of industrialization.

The preparation methods for HEA coatings mainly include laser cladding, plasma cladding, magnetron sputtering, arc spraying, electrochemical deposition, and physical vapor deposition [2]. Among them, laser cladding and magnetron sputtering are the most common methods. Because the coating produced by magnetron sputtering is too thin, its application is limited. Laser cladding is an efficient and clean surface modification technology, with the advantages of high energy density, high heating and cooling rate, less thermal impact on the substrate, and a wide range of cladding powder selection. Therefore, laser cladding technology is a suitable method to prepare HEA coatings [3].

To obtain high-performance coating materials, An Xu-long et al. [4] prepared a MoFeCrTiWAl<sub>x</sub>Si<sub>y</sub> coating on 45# steel substrate by laser cladding. Experiments showed that the addition of Si promoted the precipitation of intermetallic compounds, grain refinement, and significantly improved the hardness of coating; the maximum hardness reached 839.3 HV. However, the addition of Al inhibited the precipitation of intermetallic compounds. Therefore, the coating formed a single body-centered cubic (BCC) phase, and its hardness decreased. To further enhance the performance of HEA coatings, researchers developed a wear-reducing coating with high hardness and low coefficient of friction using a ceramic phase as the reinforcing phase. Li et al. [5] used laser cladding technology to prepare  $Ti_3Al/TiAl+TiC$  wear-resistant coating on the surface of  $Ti_6Al_4V$  alloy with Al-TiC composite powder. The weight loss due to wear of the wear-resistant coating is about 50% lower than that of titanium alloy, but the corrosion resistance decreased due to poor surface quality.

A self-lubricating composite material [6] prepared by adding a solid lubricant to HEA formed a continuous lubrication film during friction, while ensuring its rigidity and strength with a low coefficient of friction and wear rate. Moreover, solid sulfide lubricants (WS<sub>2</sub>, MoS<sub>2</sub>) have strong attachment and good film-forming properties [7]; they are one of the most widely studied and preferred solid lubricants. Based on previous study, the addition or in situ formation of a hard phase (carbide reinforcing phase) can produce a coating with better hardness and wear resistance, thus improving the coating hardness, wear resistance, and corrosion resistance.

Based on the functional positioning of coating, we designed an AlCoCrFeNi-WC-WS<sub>2</sub> cladding material system, in which WS<sub>2</sub> is a solid lubricant and WC ceramic particles form a hard-reinforced phase [8]. A functional coating with multiprincipal elements, self-lubrication, and corrosion resistance properties was prepared on a cast iron surface using preset spread powder. The effect of laser power and scan rate on the microstructure and properties of molding sample was evaluated.

# 2. EXPERIMENTAL

## 2.1. Preparation of alloy coating

Cast iron (100 mm  $\times$  75 mm  $\times$  10 mm) was used as the substrate and added to anhydrous ethanol for ultrasonic cleaning before the experiment. The cladding materials are Al, Co, Cr, Fe, and Ni HEA powders as well as WS<sub>2</sub> and WC with purity (atomic fraction) of more than 99.95% and size of 48  $\mu$ m.

The corresponding weights of Al, Co, Cr, Fe, and Ni powders were taken according to equal molar ratio and mixed evenly. At the same time, 20 Wt% WS<sub>2</sub> self-lubricating particles were added to the alloy powder as a solid lubricant, and 15 Wt% WC ceramic particles were added as a hard phase.

The powder was mixed evenly with ethanol in a grinding pot and stirred until it became a paste. The paste powder was coated on the surface of substrate using a preset powder method with a thickness of 250  $\mu$ m. An LWS-1000 laser processing equipment was selected for multichannel cladding, and in the experiment, high-purity argon (99.9%) was continuously passed at a speed of 10 L/min. The power gradient of experimental group I is 50 W, and the rate gradient of experimental group II is 60 mm/min. The experimental parameters of concrete are shown in Table 1.

Group	Laser power	Scan rate	Spot diameter	Overlap	WC	$WS_2$	Preset coating
	(W)	$(\text{mm} \cdot \text{min}^{-1})$	(mm)	rate	(wt%)	(wt%)	thickness (µm)
Ι	150-350	240	0.6	50%	15%	20%	250
Π	200	120-360	0.6	50%	15%	20%	250

Table 1. Process parameters of laser cladding

## 2.2. Methods for analysis and testing of coatings

The samples of cladding forming were cut vertically along the cladding track using wire electrical discharge machining into metal blocks of 10 mm  $\times$  5 mm  $\times$  5 mm in size, and the metal blocks were thermoset pressed on the mounting press. The pressed samples were polished in a metallographic sandpaper (280, 400, 600, 1000, 1200, and 2000 mesh) The polished mosaic samples were then polished using an MP-2A polishing machine and to mirror finish using diamond abrasion paste with a grain size of 1.5 µm. Finally, 4 Wt% nitrate alcohol corrosion solution was used to corrode the samples to observe the microscopic tissue.

An FEI-FEG quanta 250 scanning electron microscope (SEM) was used to observe the crosssection of coatings and the microscopic morphology of wear track. SEM equipped with an energydispersive X-ray spectrometer (EDS) was used to analyze the chemical composition of a microarea. A PANAlytical X'Pert Powder X-ray diffractometer (XRD) was used for phase analysis, and the parameters are as follows: Cu Ka X-ray source, acceleration voltage of 40 kV, current of 40 mA, scanning range of 10-100°, and scanning step of  $0.02^{\circ}$ /min. The microhardness of coatings was measured using a DURAMIN-40A1 automatic micro/macro hardness instrument with a load of 100 g and pressure protection time of 10 s. Friction wear experiments were conducted using a CFT-1 multifunctional material surface performance comprehensive tester with a load of 1 kg, reciprocation length of 4 mm, measurement time of 20 min, and GCr15 steel ball grinding material of  $\Phi$  6 mm. Under a laser scanning confocal microscope, the volume of wear scar, namely, the wear volume loss of coatings was precisely calculated by measuring the depth, width, and length of wear marks on the surface of coating. The samples were placed in 35 g/L NaCl solution to soak for 30 min, and then the anode polarization curve of samples in a solution was recorded at a scan rate of 10 mV/s using a CS350 electrochemical measurement system.

## **3. RESULTS AND DISCUSSION**

## 3.1 XRD phase analysis of coatings

Fig. 1 shows the XRD spectra and calibration results of composite coating prepared by laser cladding. The coating mainly shows a simple BCC structure and carbide-reinforcing phase, while other diffraction peaks are weak. This is consistent with the phase composition of HEA samples prepared by traditional vacuum melting [9]. According to Gibbs's free energy law [10], for a change in the free energy of system, the higher the hybrid entropy, the more favorable it is to reduce the free energy of system, and more favorable it is to reduce the ordering and segregation tendency of alloy. Especially, the effect of high hybrid entropy is more prominent at high temperatures, so the disordered solid solution is easier to form than the intermetallic compound during solidification. In the above phases, the carbide phase has a stronger diffraction peak at  $2\theta = 43.06^{\circ}$ . This is a typical reinforcing phase in the laser cladding layer and can significantly improve the hardness of coating. This is because the added WC particles are very small. During the cladding of coating, a part of WC is decomposed into W and C and fused into the alloy. The entry of new solute atoms makes the lattice distortion more severe. At the same time, the solubility of some solid solution of coating decreases, and other hard carbides are synthesized [11]. The spectrum shows that the intensity of diffraction peak of  $WS_2$  self-lubricating phase is relatively weaker. This is due to the decomposition of a part of WS<sub>2</sub> under the irradiation of a high-energy laser beam; WS<sub>2</sub> reacts with oxygen to generate SO<sub>2</sub> that escapes from the coating surface. The remaining WS<sub>2</sub> exists in the coating as a solid lubricant, which can effectively improve the wear resistance of coating because of its high elastic modulus and shear modulus [12].



Figure 1. XRD spectra of composite coating: (a) same laser scan rate of 240 mm/min, different laser power; (b) same laser power of 200 W, different laser scan rate

As shown in Fig. 1a, the diffraction peak intensity of BCC phase (110) mainly decreases with the increase in laser power. However, the diffraction peak intensity of carbide phase (111) crystal face gradually increases until it exceeds the BCC phase (110) crystal face and finally forms double strong peaks, indicating that the coating grows along the preferred orientation of (111) crystal face [13]. This is because the energy density of powder rapidly increases with the increase in laser power, and WC with

a very high melting point only decomposes and produces carbides with Cr and Fe under the action of high-energy laser beam. Moreover, when the laser power reaches 350 W, the diffraction peak of face-centered cubic (FCC) phase at  $2\theta = 49.94^{\circ}$  is much stronger than other parameters. This is because a very high power laser makes the Fe of substrate participate in the formation of coating [14].

# 3.2 Analysis of micromorphology and composition

a 20m 20m 20m 20m 20m 20m 20m

Figure 2. SEM images of sample with a laser scan rate of 240 mm/min: (a) 150 W, (b) 200 W, (c) 250 W, (d) 300 W, and (e) 350 W

The section morphologies of HEA composite coating made from different laser power cladding are shown in Fig. 2. As shown in Figs. 2a and 2b, when the laser power is smaller ( $\leq 200$  W), the interface between coating and substrate is significantly uneven, resulting in uneven thickness, a large number of holes in the coating structure, and accompanied with cracks and unmelted large particles. Moreover, the shape of alloy powder is irregular. The substrate and coating are mainly combined by physical function, and the coating is not firmly bonded [15]. In laser cladding, the laser provides a heat source for the formation of HEA coating; the original metal powder absorbs energy and melts, thus solidifying and

forming. The laser power determines the energy radiated per unit area, thus affecting the forming quality of alloy [16]. Therefore, the laser generated by a higher laser scan rate and lower power has a lower relative energy density, resulting in less energy absorption by alloy powder. On one hand, the low temperature inside molten pool, internal flow inadequacy, and severe spheroidization effect make the gap of spheroidization difficult to fully filled after the next powdering, thus forming pores [17]. On the other hand, the size of molten pool is small; the spacing between adjacent molten pools increases; and the powder between molten tracks is not fully melted. Therefore, the remaining unmelted powder particles are left [18].

With the increase in laser power to 250 W, no unmelted powders and cracks are present inside the coating, as shown in Figs. 2c, 2d, and 2e. Only a trace amount of small holes exist, and the coatings form favorable metallurgical bonding with the substrate. This is because thermal internal stress exists during solidification, and a large number of pores are easily derived from cracks. Thus, the amount of unmelted powder and pores inside coating gradually decreased with the increase in laser power. The increase in laser power effectively increases the size and temperature of melting pool, which in turn increases the flow inside the melting pool and improves the spheroidization inside the coating [19]. At the same time, the thickness of coating increased (Fig. 2e shows the thickest coating), and the density of structure increased.

To evaluate the composite coating and its mutual diffusion of atoms with the substrate, a linear scanning energy spectrum analysis was carried out on the cross-section of sample with a laser scan rate of 240 mm/min and laser power of 200 W. The scanning path is shown in Fig. 2b, and the result is shown in Fig. 3. As the scanning becomes closer to the surface of coating, the content of each element in the coating is balanced. Throughout scanning, the content of Fe is the highest and higher than the theoretical value, because of the high energy required by AlCoCrFeNi-WC-WS<sub>2</sub> during laser cladding to form a well-fluid melting pool. The substrate melts into a small thin layer on the surface under a very high laser beam energy, which in turn solidifies with the cladding powder. Therefore, the content of Fe in the coating increases, and the content of other elements decreases [20]. Owing to the lowest melting point of Al, when the laser beam with an extremely high temperature acts on Al instantaneously, a part of the elements are ablated and vaporized, while the other part reacts with oxygen to produce Al<sub>2</sub>O<sub>3</sub>, which is finally discharged on the coating surface in the form of scum. These lower the content of Al than the theoretical value [21]. The content of S is the lowest. Combined with the XRD analysis results, it can be further determined that some S underwent an oxidation reaction. The content of Al, Co, Cr, Ni, and S is lower near the substrate area, and then gradually increases. The content is uniform in the area near the coating surface. Near the interface, the element content shows a longer variation area from the substrate to the coating. This indicates that the diffusion of elements between the coating and substrate is obvious under laser cladding, which is beneficial to the formation of a good metallurgical combination between the coating and substrate [22].



Figure 3. EDS results of coating element components prepared at a laser scan rate of 240 mm/min and laser power of 200 W

#### 3.3 Analysis of microhardness of coatings

To explore the difference in hardness between the surface of composite coating and different positions of the substrate, the microhardness of sample cross-section was tested, and the results are shown in Fig. 4. The microhardness of coating prepared by laser cladding is 3-4 times that of substrate. The main reasons are as follows [23]: (1) The solid solution of W and C in dendrite was enhanced by the decomposition of melted WC. (2) A part of undissolved WC becomes the core of heterogeneous nucleation during nucleation. The grain nucleus increases, the grain size decreases, and the grain refinement is strengthened. (3) Fine-grain strengthening occurs by the rapid cooling of laser cladding. At the same time, the microhardness shows a gradient distribution from the base shape enhancement area to the coating surface in the sample cross-section. This is because under the same process parameters, the closer the coating surface, the faster the cooling rate of cladding coating, and the growth of the grain is limited. This is beneficial for grain refinement, so that the hardness near the coating surface is higher than that away from the coating surface position. This is because under the same process parameters, the closer the coating surface, the faster the cooling rate of cladding coating, and the smaller the crystal grains, making the hardness near the surface higher than that far away from the coating surface.

As shown in Fig. 4b and 5a, at the laser scan rate of 240 mm/min, the average hardness of each power coating is 1181.2, 1187.2, 1142.5, 1103.0, and 725.7 HV. The average hardness of coating first increases and then decreases with the increase in laser power. The hardness of coating significantly decreases to 725.7 HV when the power reaches 350 W, 40% lower than the maximum hardness at 200 W. This is because the laser power will directly affect the energy density of the coating unit area. When the laser power is smaller ( $\leq$ 200 W), the powder melts fully; the number of cracks and pores within the coating decreases; and the content of in situ synthesized chromium carbide and iron carbide hard-reinforced phase increases accordingly [24].When the laser power exceeds 200 W, the laser energy density is too high, resulting in too much melting of the substrate; i.e., the dilution rate increases, so that a large amount of substrate elements participates in the formation of coating and decreases the hardness of coating. At this time, FCC phase plays the main role in the coating structure, and the cooling rate of

cladding layer significantly decreases, thus producing coarse grains and significantly decreasing the coating hardness [25].

As shown in Fig. 4c and 5b, when the laser power is constant 200 W, the average hardnesses of the coating corresponding to different scan rates are 1036.2, 1126.5, 1187.2, 1092.5, and 1089.3 HV. With the increase in scan rate, the hardness increases slowly and then decreases gradually. When the laser scan rate is less than 240 mm/min, the powder is subjected to a larger density of laser energy per unit time, a larger melting pool size, and a higher melting and dilution rate of substrate, and some substrate elements participate in the formation of coating, making the hardness of coating slightly lower than the maximum value. As the laser scan rate increases from 240 mm/min to 360 mm/min, the cooling rate increases the stress, cracks, and pores inside the coating [26], decreasing the hardness of coating.



**Figure 4.** Microhardness of composite coating: (a) SEM image of hardness testing of coating; (b) same laser scan rate of 240 mm/min, different laser power; (c) same laser power of 200 W, different laser scan rate

## 3.4 Analysis of wear resistance of coatings

Fig. 5 shows that with the increase in laser power or scan rate, the wear volume loss of coating first increases and then decreases in different degrees. In other words, the wear resistance first decreases and then increases, negatively correlated with the variation trend of average hardness of coating, consistent with the classical Archard law of wear [27]. The minimum wear volume loss of coating  $(18.412 \times 10^{-3} \text{ mm}^3)$  is much smaller than that of substrate  $(53.9 \times 10^{-3} \text{ mm}^3)$ , indicating that the wear resistance of coating is better than that of substrate. This is because a rigid secondary phase is formed by the fine hard carbide particles in the coating microstructure. This transfers the effect during friction wear and is not easy to be ploughed by grinding particles, thus reducing the amount of sample wear [28]. During friction, because of the loading force, the solid lubricant WS<sub>2</sub> at the protruding part of coating surface softens and transferred to the concave part to form a discontinuous lubrication film [29].



**Figure 5.** Average hardness and wear volume loss of composite coating: (a) same laser scan rate of 240 mm/min, different laser power; (b) same laser power of 200 W, different laser scan rate

Fig. 6 shows the SEM image and EDS energy spectrum analysis results of surface morphology of coating after wear. As shown in the picture, furrow and debris appeared on the surface of coating. This is due to a change in wear mechanism: The initial separation layer wear of coating is transferred into the abrasive wear [30], so the wear surface is rough. According to the results of EDS analysis, on the surface of worn coating, the content of C and W is relatively high, and some S and O are present. However, compared with the HEA coating without solid lubricants and ceramic particles, the surface oxidation of AlCoCrFeNi-WC-WS<sub>2</sub> composite coating decreases. Also, the furrow is shallow, deciduous debris is less, and wear surface is relatively flat. This is because the addition of WC increases the number of in situ synthesis of hard carbide phases in the coating, improves the effect of dispersion strengthening, and plays certain resistance role to the embedding and reciprocating motion of grinding ball during friction [31]. Another important factor is that an appropriate amount of WS<sub>2</sub> is present in the coating as self-lubricating phase, shows favorable wear resistance, and ensures the formation and stability of lubrication film during friction wear [32].



**Figure 6.** Surface morphology after wear of coating prepared by a laser scan rate of 240 mm/min and the laser power of 200 W

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#### 3.5 Analysis of corrosion resistance of coatings

To explore the corrosion resistance of composite coating and analyze the effect of laser power and scan rate on the corrosion resistance of coating, all the samples and substrate were placed in 3.5 Wt% NaCl solution for electrochemical test. Fig. 7 shows the anodic polarization curve measured by soaking the samples in a corrosion solution for 30 min. As shown in the polarization curve in Fig. 7, the passivation range of coating prepared under different process parameters is the same, while the passivation range is larger than that of substrate. According to the electrochemical theory [33], the interface of passivation layer and solution can adsorb harmful Cl<sup>-</sup> in corrosion liquid containing chloride, and oxide film is penetrated by Cl<sup>-</sup>. Also, the larger the coating passivation zone of polarization curve and the stronger the ability of hindering Cl<sup>-</sup> penetration, the pitting corrosion resistance ability is better. This is because a compact passive film is formed by the corrosion-resistant element Co, Cr, and Ni inside the coating, thus strengthening the corrosion resistance of coating. In addition, the passivation area of the coating appears significantly earlier [34], indicating that it has better pitting-resistance property.



**Figure 7.** Potentiodynamic polarization curves of composite coating: (a) same laser scan rate of 240 mm/min, different laser power; (b) same laser power of 200 W, different laser scan rate

After the polarization curve of composite coating was obtained, the polarization curve was fitted using the cathodic Tafel curve extrapolation method, and the fitting results are shown in Table 2. According to electrochemical theory [35], the smaller the corrosion current density ( $I_{corr}$ ), or the larger the corrosion potential ( $E_{corr}$ ), the stronger the corrosion resistance of material; i.e., the smaller the corrosion current density ( $I_{corr}$ ), the slower the corrosion rate (CR). Table 2 shows that the  $I_{corr}$  of coating fluctuates up and down by many orders of magnitude with the increase in laser power. When the laser power is 200 W, the  $I_{corr}$  of coating is lower than that of substrate, and its  $E_{corr}$  is higher, indicating that the corrosion resistance of coating is stronger than that of substrate and is optimal. However, when the laser power is 350 W, simultaneously the  $I_{corr}$  and  $E_{corr}$  of coating are the lowest. In combination with Fig. 2e, it is observed that the coating is thicker, and fewer internal cracks and pores are present at this condition, thus reducing the CR. However, as more substrate elements such as Fe participate in the forming process of coating, the FCC phase plays the leading role in forming the coating first decreases and then increases with the increase in laser scan rate. When the scan rate is the lowest of 240 mm/min, the

 $E_{corr}$  is high, indicating that its resistance to uniform corrosion is obviously better than that of substrate and coatings prepared by other parameters. Therefore, its corrosion resistance is the best.

Parameter	$I_{corr}(\mu A \cdot cm^{-2})$	$E_{corr}(mV)$	$CR(\mathbf{mm}\cdot\mathbf{a}^{-1})$
Substrate	9.836	-789.9	0.1571
240 mm/min, 150 W	23.548	-596.5	0.2581
240 mm/min, 200 W	1.576	-663.8	0.0192
240 mm/min, 250 W	9.136	-779.5	0.1140
240 mm/min, 300 W	15.791	-452.2	0.1911
240 mm/min, 350 W	3.207	-723.4	0.0388
200 W, 120 mm/min	11.933	-561.3	0.1444
200 W, 180 mm/min	3.365	-717.5	0.0407
200 W, 240 mm/min	1.576	-663.8	0.0190
200 W, 300 mm/min	4.971	-461.1	0.0742
200 W, 360 mm/min	27.132	-558.4	0.3283

Table 2. Corrosion parameters of composite coating

#### **3. CONCLUSIONS**

1) Using laser cladding technology, a composite coating of AlCoCrFeNi-WC-WS<sub>2</sub> was prepared successfully. The surface quality is more superior than the traditional HEA, and the best process parameters are as follows: laser power of 200W and scan rate of 240 mm/min.

2) The composite coating AlCoCrFeNi-WC-WS<sub>2</sub> is mainly composed of an FCC phase, a BCC phase, a carbide-reinforcing phase, and a slight amount of WS<sub>2</sub> self-lubricating phase. The composite carbide is composed of WC and in situ synthesized chromium carbide and iron carbide.

3) The composite coating AlCoCrFeNi-WC-WS<sub>2</sub> was prepared using laser cladding technology. Owing to the addition of WC particles, its hardness increased by 3-4 times of the substrate. Although a part of WS<sub>2</sub> decomposes, a part of the WS<sub>2</sub> self-lubricating phase remains in the coating, provides great lubrication, and enhances the wear resistance. At the same time, the coating shows favorable corrosion resistance under the synergy effect of the carbide-reinforcing phase and WS<sub>2</sub> self-lubricating phase.

#### **ACKNOWLEDGEMENTS**

The project was supported by China Postdoctoral Science Foundation (2018M632313) and the Open Fund for Jiangsu Key Laboratory of Advanced Manufacturing Technology (No. HGAMTL-1710).

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