Interfacial Aspects of Metal Matrix Composites Prepared from Liquid Metals and Aqueous Solutions: A Review

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Received: 26 September 2020; Accepted: 20 October 2020; Published: 21 October 2020

Abstract: The paper reviews the preparation of the different metallic nanocomposites. In the preparation of composites, especially in the case of nanocomposites, interfacial phenomena play an important role. This review summarizes the literature on various interfacial phenomena, such as wettability and reactivity in the case of casting techniques and colloidal behavior in the case of electrochemical and electroless methods. The main contribution of this work lies in the evaluation of collected interfacial phenomena and difficulties in the production of metal matrix composites, for both nano-sized and micro-sized reinforcements. This study can guide the composite maker in choosing the best criteria for producing metal matrix composites, which means a real interface with good adhesion between the matrix and the reinforcement. This criterion results in desirable mechanical and physical properties and homogenous dispersion of the reinforcement in the matrix.

Keywords: metal matrix composites; wettability; Al matrix; Ni-P matrix; zeta potential

1. Introduction

Metal matrix composites are made up of a metal matrix and a reinforcing substance, which may be metal or another substance. Composites can be classified in various ways; some examples are formation type, preparation method and matrix type. According to the formation of the composites, we can group them into in-situ [1] and ex-situ composites [2]. The reinforcement could be graphite [3], graphene [4–9], carbon nanotubes [10–13], ceramics [14–17], metallic nano-sized particles [18–20] or fibers, whereas the matrix materials could be aluminum [21,22], magnesium [23], steel [24], copper [25], etc.

According to the preparation method [26,27], we can group composites into stir casting [28–32], assisted stir casting [33], pressure infiltration [34–37], pressureless infiltration [38], laser melting [39], electrochemical [40], electroless deposition [41–43] and powder metallurgy [44–50] methods. Based on the physical state of the matrix material, we can form three groups, namely the molten matrix (in the case of stir casting, pressure infiltration, pressureless infiltration, laser melting), the aqueous or non-aqueous solution of the matrix element (electro- and electroless deposition) and solid phase matrix (powder metallurgy). The methods and interfacial phenomena of metal matrix composite preparation are shown in Figure 1.

During the preparation of nanocomposites (using classification by the physical state of the matrix), the interfacial phenomena have the most important role. The following sections show the effect of interface on the structure of nanocomposites prepared by casting and aqueous methods.
Figure 1. Methods and interfacial phenomena of metal matrix composite preparation.

2. Using Melted Metal Matrix for the Preparation of the Composite

2.1. Role of the Wettability of the Reinforcement by Molten Metal

In metal matrix nanocomposites (MMNCs), the second ceramic phase is used as the reinforcing phase. This may have a particulate, fibrous or needle structure and at least one of the characteristic dimensions is less than 100 nm. The consequence of the size of the reinforcement is that in the manufacturing of MMNCs, interfacial phenomena—forces [51,52] and energies [53]—play a decisive role in the interaction of the reinforcement and the (molten) matrix metal. In the nanoscale range, the gravitational force and the difference in density between the melt and the particle no longer affect the subsidence of the particle. In such a case, the interfacial phenomena acting between the components influence the movement, penetration and incorporation of the reinforcement into the matrix. As a result of these interfacial interactions, the structure of the composite is formed, which decisively determines the strength and mechanical properties of the composite materials.

Based on thermodynamic formulations, we can derive the necessary criteria as appropriate parameters, including the interfacial energies [54]. The contact angle is characteristic of the interfacial behavior of solid–liquid phases when they are in contact. It is best observed when a small drop of molten metal is placed on the surface of a flat solid ceramic. The molten metal then assumes a characteristic shape, which may be spherical, hemispherical or fully spread, depending on the surface tension and adhesion energy prevailing in the system. The formed drop shape can be characterized by the contact angle, which is the angle between the tangent to the contour of the liquid drop at the point of contact of the liquid, the solid phase and the gas phase (the “triple line”). The contact angle is the angle between the tangent and the solid phase in the direction of the liquid phase (Figure 2).
Young’s equation is valid between the contact angle and the interfacial energies:

$$\cos \theta = \frac{\sigma_{cv} - \sigma_{cl}}{\sigma_{lv}}$$  \hspace{1cm} (1)

where

- $\sigma_{cv}$—the surface energy between the ceramic and the vacuum, J/m$^2$
- $\sigma_{cl}$—the interfacial energy between the ceramic and the melt, J/m$^2$
- $\sigma_{lv}$—the surface tension of the melt, J/m$^2$.

Equation (1) is significantly simplified if we take into account the so-called adhesion energy, which is by definition:

$$W = \sigma_{cv} + \sigma_{lv} - \sigma_{cl}$$  \hspace{1cm} (2)

Adhesion energy is always a more positive number than 0; its unit is also J/m$^2$. The higher the value of $W$, the stronger the mutual attraction between the molten metal and the ceramic, i.e., the adhesion. In the production of metal–matrix composites, it is theoretically worthwhile to use only metal–ceramic pairs that stably form two phases in contact with each other, i.e., for whom the value is certainly positive. Thus, for cermet pairs used in the production of metal matrix composites, the adhesive energy has the following range:

$$0 < W < \sigma_{cv} + \sigma_{lv}$$  \hspace{1cm} (3)

Substituting Equation (2) into Equation (1) gives the following relation for the contact angle:

$$\cos \theta = \frac{W}{\sigma_{lv}} - 1$$  \hspace{1cm} (4)

The value of the contact angle can be in the range of 0–180°, whose values as well as the ratios of energies clearly characterize the wetting conditions prevailing in the system. Based on (4), different ranges of the contact angle can be determined:
(a) $\geq 90^\circ$: the melt does not wet the ceramic; the adhesion energy is less than the surface tension of the melt;
(b) $<90^\circ$: the melt wets the ceramic; the adhesion energy of the melt surface is greater than the surface tension of the melt (but less than twice the surface tension);
(c) $=0^\circ$: the melt completely wets the ceramic; the adhesion energy is at least twice the surface tension of the melt.

One of the crucial points in the production of casting composites is the introduction of the reinforcing phase into the molten metal. Perfect wettability between reinforcement and the molten metal is required for complete immersion of the reinforcement [55–58]. If the molten metal does not perfectly wet the reinforcement phase, the composite can only be produced by prolonged stirring [59]. Hashim et al. [59] made the following observations during the preparation of die-cast aluminum composites:

- stirring does not promote the immersion of the particles in the matrix: the particles float on the surface of the molten metal, regardless of the speed of stirring;
- upon stirring, the ceramic particles are incorporated into the solidifying metal, but during the remelting of the composite, when the metal is completely melted, the composite can be separated;
- using magnesium improves wetting, but if the magnesium content of the aluminum melt exceeds 1 wt.%, the viscosity of the melt increases.

Another crucial point in the production of casting composites is the agglomeration of the reinforcement [60]. The cause of coagulation of the reinforcement can also be traced back to wetting, i.e., the less the molten metal wets the ceramic surface, the more likely the ceramic particles are to adhere to each other. Avoidance of this unfavorable process, and thus the homogeneous distribution of the reinforcement in the molten metal, can be achieved if the contact angle of the ceramic–molten metal pair is as close as possible to $\theta = 0^\circ$.

In general, one can say that the surface of the frequently used reinforcement is not perfectly wetted by molten metals. To achieve perfect wetting and perfect dispersion of the particles in the matrix, various modifications—for example, coatings or auxiliary phases—must be used in these systems. Another alternative is to modify the production method—for instance, by using an ultrasonic assisted application [60].

Two typical examples of composites for non-wetting and perfect wetting systems are shown here. The left-hand picture in Figure 3 shows non-wetting, where there is no adhesion and/or no common interface between the matrix and the reinforcement; here, there is no connection between the C fiber and the matrix. The right-hand picture shows the perfect wetting state; there are no pores on the matrix/reinforcement interface, as the molten matrix is able to flow around the reinforcement fibers or particles.

2.2. Improvement of the Wetting Behavior

The wettability of the reinforcement by molten metal can be improved by the modification of the surface of the reinforcement, e.g., by formation of a metallic coating on it. Another opportunity is the application of a molten salt phase, which can prevent the molten metal from further oxidation and can help in the formation of a well-wetted coating on the surface of the reinforcement.
Improvement of the wetting behavior of the liquid metal can be achieved by the modification of the oxide layer present on the surface of the reinforcement. Generally, one can say that molten metal or molten metal/metal-like ceramic systems does not wet the surface of the reinforcement. Generally, one can say that molten metal wets well the surfaces of a solid metal or metal-like ceramics. In the case of molten metal/solid metal or molten metal/metal-like ceramic systems, the contact angle is lower than $90^\circ$, typically between $0^\circ$ and $50^\circ$. During the selection of the metal for the coating, the main consideration is that the metal providing the coating should be an alloy of the matrix and not form brittle or undesirable phases with the matrix.

In the production of metal matrix composites, the surface of the reinforcement is most frequently covered with a copper or nickel coating [61]. Electroless nickel and copper coatings can be prepared on the surface of metals [62,63], carbon fiber [64–67], $\text{Al}_2\text{O}_3$ surface [68] and SiC particles [69–74]. As with metals, metal-like ceramics such as TiC can be used as coatings [75].

The contact angle of molten aluminum on an SiC surface is $\theta = 150^\circ$; this contact angle is reduced to $\theta = 115^\circ$ at 2 h holding time. The contact angle of the molten aluminum on a Ni-coated SiC surface is $\theta = 12^\circ$. The nickel coating also prevents the formation of $\text{Al}_4\text{C}_3$ on the Al/SiC interface [68]. The wetting behavior of different solid materials by a molten matrix is tabulated in Table 1. Perfect wettability or good wetting is the criterion of the preparation of the metal matrix composites from liquid metal. One can see from Table 1 that to reach good wetting behavior of the liquid metal, the oxide layer needs to be removed (e.g., in the case of Al) and one needs to choose a metal or a metal-like ceramic required to be coated on the surface of the reinforcement.

![Figure 3](image-url)

**Figure 3.** The reinforcement (C-fiber) and the matrix (aluminum) position in the case of a “non-wetting” and a “perfect wetting” system.

### 2.2.1. Modification of the Surface of the Reinforcement

Due to the difference in the chemical bonding between the matrix metal and the reinforcement, the molten metal does not wet the surface of the reinforcement. Generally, one can say that molten metal wets well the surfaces of a solid metal or metal-like ceramics. In the case of molten metal/solid metal or molten metal/metal-like ceramic systems, the contact angle is lower than $90^\circ$, typically between $0^\circ$ and $50^\circ$. During the selection of the metal for the coating, the main consideration is that the metal providing the coating should be an alloy of the matrix and not form brittle or undesirable phases with the matrix.

| Liquid Phase | Solid Phase | Temperature | Contact Angle | Reference |
|--------------|-------------|-------------|---------------|-----------|
| Al           | $\text{Al}_2\text{O}_3$ | 700 °C       | 140°          | [76]      |
| Al (oxide layer free) | $\text{Al}_2\text{O}_3$ | 710 °C       | 63°           | [77]      |
| Al           | Zr          | 700 °C       | $\sim 10^\circ$ | [76]      |
| Al           | 670 nm thick Zr-coated alumina | 700 °C       | 20°           | [76]      |
| Al (oxide layer free) | $\text{SiO}_2$ | 710 °C       | 23°           | [77]      |
| Al (oxide layer free) | TiB$_2$ | 710 °C       | 0°            | [77]      |
| Al (oxide layer free) | TiB$_2$ | 710 °C       | 10°           | [77]      |

**Table 1.** The wettability of the different materials by molten metal matrix.
Table 1. Cont.

| Liquid Phase | Solid Phase       | Temperature | Contact Angle | Reference |
|--------------|-------------------|-------------|---------------|-----------|
| Al           | Graphite          | 740 °C      | 140°          | [78]      |
| Al           | Ni                | 740 °C      | 45°           | [78]      |
| Al           | Ni-coated graphite| 740 °C      | 27°–45°       | [78]      |
| Cu           | WC                | 1080 °C     | 25°           | [79]      |
| Cu           | WC                | 1133 °C     | 0°            | [77]      |
| Cu           | WC-Co             | 1080 °C     | 6°            | [79]      |
| Cu           | Graphite          | 1227 °C     | 140°          | [80]      |
| Cu           | Cr3C2             | 1227 °C     | 0°            | [80]      |
| Zr2Cu        | WC                | 1150 °C     | 28°           | [81]      |
| Ni           | B4C               | 1480 °C     | 102°          | [82]      |
| Ni           | ZrC               | 1480 °C     | 73°           | [82]      |
| Ni           | TiC               | 1480 °C     | 25.5°         | [82]      |
| Ni           | SiC               | 1480 °C     | 104°          | [82]      |
| Ni-based superalloy | Al2O3-based | 1500 °C | 141° | [83] |
| Ni-based superalloy | SiO2-based | 1500 °C | 143° | [83] |
| Ni-based superalloy | ZrSiO4 | 1500 °C | 136° | [83] |

2.2.2. Molten Salt-Assisted Process

Roy et al. [84] investigated the effect of the addition of chloride and fluoride salts to a basic equimolar NaCl-KCl mixture, examining the coalescence of small droplets. Coalescence can occur after the removal of the oxide layer from the molten aluminum droplets. A perfect oxide removal process, i.e., good coalescence, was achieved in the case of fluoride salt addition. The best additives are KF and NaF, LiF, Na3AlF6. One can say that the smaller the Al/salt interfacial energy, the more intensive the oxide removing effect. The same observation has been made in the case of NaCl-KCl-K2TiF6 salt mixture. Under the melted NaCl-KCl-K2TiF6 salt, the molten aluminum can perfectly wet the surface of a graphite [85,86]. The perfect wettability of aluminum on graphite under molten salts is achieved through the next steps:

1. The oxide layer is dissolved from the surface of molten aluminum due to the K2TiF6. In the molten salt, an oxo-fluoro complex compound is formed which prevents the further reaction of Ti ions.
2. As the molten salt/aluminum ratio increases, the Ti content increases in the system (salt/aluminum/graphite). Due to higher Ti content, the free Ti ion number will be raised so the possibility of the exchange reaction also grows.
3. At a critical Ti content and temperature (higher than 0.4 w% Ti in aluminum and above 750 °C), TiC can be formed at the Al/C interface. Due to the TiC nanolayer at the interface, the wettability of graphite by molten aluminum will be increased.
4. During the cooling of the sample, an Al3Ti intermetallic phase will be created due to a decrease in the solubility of Ti.

The wettability of TiC by molten aluminum was investigated by Lopez et al. [87] under Ar gas and molten KF-AlF3 eutectic mixture. They observed that the aluminum does not wet the TiC under Ar but perfectly wets that under molten salt. The role of the molten salt was to clean the surface of Al and TiC of the oxide layer. This observation proves that if one can prepare a TiC coating on the carbon surface, perfect wettability could be achieved. Kennedy and Karantzalis [88] investigated the effect
of KAlF$_4$ and K$_3$AlF$_6$ salts on the incorporation of the reinforcement particles in molten aluminum. They observed that the graphite particles have not been incorporated in molten aluminum. From these two observations, we can determine that the oxide layer removal alone is not enough to reach perfect wettability. In addition to the fluoride ion responsible for decomposing the oxide, the molten salts must contain an ion capable of forming a coating on the surface of the reinforcement which can be perfectly wetted by the molten aluminum.

K$_2$TiF$_6$, as mentioned above, is used as an additive to the basic salt, but one can use it as the pure salt as well. Using pure K$_2$TiF$_6$, the reinforcement is mixed with the salt and then this mixture is admixed to the molten aluminum. Another possibility is to prepare a saturated aqueous solution of the K$_2$TiF$_6$ at around 100 °C. The reinforcement is admixed with the saturated solution and then the salt is dried and crystallizes on the surface of the reinforcement, as mentioned in [89] for the case of Al/C, Al/SiC composites.

Lekatou et al. [2,90] prepared an aluminum matrix composite reinforced with TiC and WC nanoparticles using K$_2$TiF$_6$ salt. In their experiments, the salt and carbide particles were mixed and then introduced into the aluminum melt with vigorous stirring. Stirring ensured a homogeneous distribution of the reinforcement phase. Al$_3$Ti and Al$_5$W intermetallic phases were also identified in the aluminum matrix composite using WC particles.

Reactive salts are able to form in-situ metal matrix composites as well. In this case, the reinforcement will be formed due to a chemical reaction between the salt and a component of the matrix or other additives. In the molten salt-assisted in-situ method, (nano) composites can be prepared using different reactive salts [91]. In this procedure, Birol prepared a TiC-reinforced aluminum matrix composite [92], while Mahamani developed in-situ TiB$_2$/ZrB$_2$-reinforced aluminum matrix composites [93]. Other salt-assisted in-situ reinforcements could include TiB$_2$ [94], TiN nanoparticles [95] or Al$_3$Ti [96].

3. Composite Preparation from Aqueous Solutions

Composite coating, especially Ni and Ni alloy matrix coating, can be prepared by electrodeposition [97–101] or electroless [102–109] methods. Electrodeposition, in contrast to electroless deposition, forces the deposition process on the surface of the cathode using potential difference. The electroless method means that a reducing agent can be used instead of voltage [43]. The potential reducing agents are hydrazine [110], sodium hypophosphite (NaH$_2$PO$_4$) [111,112], sodium-borohydride (NaBH$_4$) [111]. One type of highly investigated electroless Ni composite coating is the phosphorus-containing Ni-P, so the next part of the paper focuses on such composites.

Ni-P coatings prepared by deposition from aqueous solution have good mechanical properties. These can be improved by subjecting the product to heat treatment, during which nano- or micro-Ni$_3$P phases can appear. In this case, one can actually speak of an in-situ composite in which the reinforcement is the second phase formed during the heat treatment. The properties of Ni-P coatings can be further modified by the co-deposition of a well-chosen reinforcement (ex-situ composite). In addition to improving hardness and wear resistance, the presence of a second reinforcing phase also improves the corrosion resistance of the Ni-P coatings [113,114].

Ni-P matrix composites are reinforced using different nano- and microparticles, of which the most common are SiC [115], SiO$_2$ [116], TiO$_2$ [117], ZrO$_2$ [118], WC [119] and, more recently, TiC [120]. Furthermore, graphene [121] and nano-diamonds also appear in the literature as a reinforcement [80].

Initial attempts to prepare Ni-P composites failed, as described in the literature. This is because nickel baths very often become unstable due to the high specific surface area of the reinforcement. The unstable bath disintegrates, i.e., the Ni$^{2+}$ ions precipitate on the surface of the particles in the form of some Ni compounds. Various stabilizers are added to the bath to prevent the bath from disintegrating.
Thiourea [133,134], Pb-acetate and maleic acid [135] have been used as stabilizers. Due to the stabilizers, it has become possible to produce Ni-P matrix composites by reduction [136]. One of the reasons for the disintegration of the bath is the electrokinetic behavior of the reinforcement, which is characterized by the zeta potential. The zeta potential in the Ni-P bath-reinforcement dispersion can be modified by the addition of surfactants.

A prerequisite for the production of Ni-P matrix composite coatings with a uniform distribution of the reinforcement and the designed mechanical properties is that the reinforcement phase can be homogeneously dispersed in the Ni-P bath, in both the electro- and electroless deposition methods. To avoid the agglomeration of the particles in the bath, there is a crucial physico-chemical property, namely the zeta potential of the particles in the bath [43,123].

Nano-sized particles easily form agglomerates due to their high specific surface area, zeta potential and wetting properties. Ensuring the proper dispersion of the nanoparticles in the bath can be achieved by the continuous stirring of the bath, sonication treatment, or using surfactants. Regarding the surfactant, cationic, anionic and non-ionic forms are used in practice. As a result of these surfactants, the bath can wet the surface of the reinforcement, and they can also modify the zeta potential value. In the case of increasing the value of the zeta potential by changing the composition and pH of the bath, the particles push away from each other to a greater extent, thus preventing their agglomeration [136,137].

In connection with this, various studies have been carried out in which the properties of the Ni-P bath–particle system were investigated by measuring the zeta potential and the study of the particle size distribution, deposition and the amount of reinforcement in the composite.

Tamilarasan et al. [138] deal with the development of a Ni-P-TiO\textsubscript{2} composite coating. They observed that as the surfactant concentration increased, the amount of TiO\textsubscript{2} particles in the Ni-P-TiO\textsubscript{2} composite varied according to a maximum curve. It was also observed that when using a cationic DTAB (Dodecyltrimethylammonium Bromide) surfactant, the amount of TiO\textsubscript{2} nanoparticles in the composite was twice the value of SDS. The changing of the amount of the reinforcement in the composite according to a maximum curve is described for Al\textsubscript{2}O\textsubscript{3} particles using a cationic surfactant [139].

Using CTAB (Cetyltrimethylammonium Bromide), Liu et al. [137] developed Ni–P–Al\textsubscript{2}O\textsubscript{3} composite coatings. The cationic surfactant prevented the Al\textsubscript{2}O\textsubscript{3} particles from agglomerating in the bath. In parallel, however, it was found that the proportion of Al\textsubscript{2}O\textsubscript{3} in the coating decreased significantly with increasing CTAB concentration. In their work, they conclude by stating that the stability of the sol formed by the bath and Al\textsubscript{2}O\textsubscript{3} is due to the fact that CTAB changes the zeta potential of the particle to a positive value. This finding was not supported by measurements. Comparing these results with the results of Necula et al. [140], it can be clearly seen that the measured zeta potential of Al\textsubscript{2}O\textsubscript{3} particles in the nickel bath shows a decreasing trend as a function of the pH range studied by Liu [137] as well. The zeta potential drops from the initial 10 to −2 mV. Comparing these results with the zeta potential value measured in the sol formed by distilled water- Al\textsubscript{2}O\textsubscript{3}, which is 50 ± 2 mV, it can be seen to what extent the presence of Ni\textsuperscript{2+} in the bath deteriorates the zeta potential value and thus the stability of the sol. The 10−(-)2 mV zeta potential measured by Necula explains the formation of agglomeration of the particles in the bath, which was also observed by Liu et al. [137].

In the case of Ni-P-TiC composite preparation, it has been reported that the use of an anionic surfactant (linear alkylbenzene sulfonate; LABS) results in the most TiC in the composite, but the nanoparticles are in the composite as agglomerations. In comparison, with the use of polymeric (polyethylene glycol) and cationic (CTAB) surfactants, the particles did not agglomerate, but the amount of TiC deposited lagged behind the results obtained with the anionic surfactant. Together with this observation, it was found that in the anionic case, the separated structure was composed of many spherical (cauliflower) units. This is because the higher amount of TiC provides more nucleating sites for the deposited Ni-P matrix [141,142].

In many cases, it has been published in the literature that the layer thickness of the deposited composite decreases as the concentration of the surfactant in the bath is increased. One reason for this
may be that surfactants reach the surface of the liquid and reach the substrate, which also acts as a catalyst, sooner than Ni$^{2+}$ ions coming from inside the solution. An additional reason may be that by increasing the concentration of the surfactant, in addition to the decrease in the surface tension of the bath, the formation of micelles [43] must also be expected, which may prevent the escape of hydrogen gas during reduction. During reduction deposition, hydrogen gas near the surface of the substrate also inhibits the deposition of Ni$^{2+}$ ions on the substrate [143]. One can observe a correlation between the wetting angle ($\theta$) measured on the surface of the particles and the zeta potential ($\zeta$) based on data in the literature [43,144,145].

4. Conclusions

According to the physical state of the matrix, we can group the metal matrix composite methods as follows: techniques using a molten matrix, powder metallurgy and aqueous solution-assisted methods. The interface between the reinforcement and the matrix plays a crucial role. The most important physicochemical parameters are wettability and reactivity, and in the case of the aqueous method, electrokinetic behavior, i.e., the zeta potential. In the casting method and powder metallurgy, it is a well-known fact that the surface of the matrix is covered by an oxide layer. The first step in these methods is to remove the oxide layer or avoid its formation. Using a melted matrix, the second step is modifying the surface of the reinforcement to avoid the chemical reaction between the reinforcement and the matrix and reach better wettability (closer to $\theta = 0$). In the third group, using aqueous solutions, one needs to calculate the zeta potential, which correlates to the contact angle of the reinforcement in the aqueous solution. Zeta potential is responsible for the homogeneous dispersion of the reinforcement in the bath (aqueous solution) and also in the matrix.

**Funding:** This research received no external funding.

**Acknowledgments:** The research work presented is based on the results achieved within the GINOP-2.3.2-15-2016-00027 “Sustainable operation of the workshop of excellence for the research and development of crystalline and amorphous nanostructured materials” project implemented in the framework of the Szechenyi 2020 program. The realization of this project is supported by the European Union.

**Conflicts of Interest:** The author declare no conflict of interest.

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