

Original Research

Physical Distribution of Nanoparticles by Ultrasonic Operation

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ABSTRACT

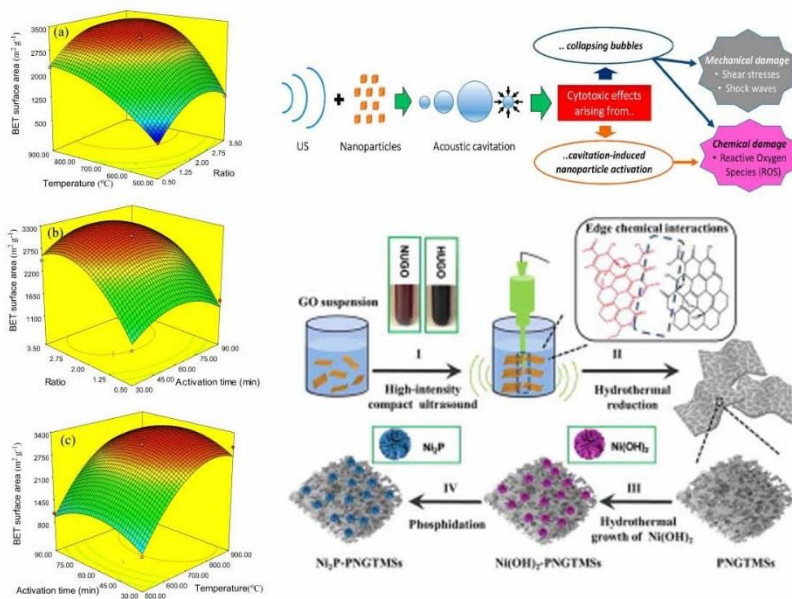
In this method, particles are separated from each other using destructive energies such as ultrasonic vibrational energy. The propagation of ultrasonic waves in solution produces very large local pressures thousands of times the atmospheric pressure, which breaks the bond from clumping. Changing the pH of the plating bath changes the zeta potential of the particles. Zeta potential is the electric potential that exists at the boundary between the solid surface and the liquid environment in which the solid particles are located and is a function of the surface charge of the particle, the adsorbed layer at the interface, the type and composition of the medium in which the particle is suspended. The magnitude of the zeta potential of a particle is a measure of the interaction of particles in such a way that it can be used to predict the long-term stability of the solution. If the particles have a positive or negative charge, they tend to repel each other and resist agglomeration. On the other hand, if the particles have low zeta potential, there is no factor to prevent the particles from agglomerating and as a result, the particles become agglomerated. The boundary between stable and unstable solutions is +20 mV and -20 mV, which means that if the zeta potential of the solution is more than +20 mV or less than -20 mV, the solution is usually stable, and if the zeta potential is between 20+ mV and -20 mV, there are unstable solutions and agglomerated particles.

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GRAPHICAL ABSTRACT



Introduction

Chemical Methods of Dispersion of Colloidal Systems

In this method, by adding surfactants, by reducing the ionic strength of the bath and by changing the pH, the diameter of the agglomerates can be reduced [1-5].

A. Change in pH

Table 1. Stable conditions in terms of zeta potential

Colloidal Stability	Average zeta potential (MV)
From infinite stability to very good stability	-100 to -60
Reasonable stability	-60 to -40
Medium stability	-40 to -30
Poor dispersion threshold	-30 to -15
Bonding threshold	-15 to -10
Intense bonding and deposition	-5 to +5

Most substances have zeta potential when suspended in water. Zeta potential is affected

by factors such as pH, additive concentration and ionic strength of the solution. The most important factor that affects the zeta potential is pH [6-8]. The zeta potential of many chemical surfaces, such as mineral oxides in dilute salt solution, show a certain behavior by changing the pH, so that when the pH increases with the addition of alkalis, the zeta potential becomes more negative or at least its positive value decreases. By adding acidic substances, their ionization reduces the number of hydroxyl ions and thus the particle surface becomes more positive. In the zeta potential curve in terms of pH, there are pHs where the curve crosses the zero potential. This point is called the isoelectric point¹. This point indicates the pH value at which the solution has the lowest stability at pH [9-12]. The use of pH changes to reduce the diameter of the lumps should be done with caution as these changes

may cause deterioration of the quality of the coating [13-18].

B- Use of surfactants

When surface activator agents or surfactants are immersed in emulsion solutions or mixtures and suspension, they have a strong tendency to form adsorption common chapters. Hence, they reduce surface tension, wetting and dispersing. Surfactants are an organic molecule that has a water-soluble part (hydrophilic) and a fat-soluble part (hydrophobic). According to the structure of surfactant, they are divided into 4 main types: anionic, cationic, non-ionic and amphoteric [19-22]. In anionic ions, the water-soluble part has a negative electric charge, while their cationic types have a positive charge. Non-ionic surfactants are uncharged and amphoteric surfactant has positive and negative charges on each part of the molecule. Surfactants disperse particles by increasing the electric potential (electric charge) of the surface of the particles [23-26]. By using surfactants that have electron-rich aromatic rings and a flat molecular structure, by absorbing them much more easily or by creating a barrier of organic materials around the particles, particle aggregation is dramatically avoided and even completely prevented. The transparency of the solution is a critical measure of colloidal stability and is determined by ocular observation. Depending on the concentration of surfactant, different states are seen in the transparency of the solution [27-30]. In Table 2 at very low concentrations of surfactant, the

particles coagulate and the particles are seen as floating masses. At somewhat higher concentrations, although the concentration of the surfactant is still very low, the solution appears opaque or cloudy. With increasing concentration of the surfactant, the cloudy solution gradually turns into a milky solution, which typically has colloidal stability conditions. At higher concentrations the solution will become completely transparent [31-35].

Table 2. Different conditions of solution transparency according to surfactant concentration

surfactant concentration	solution transparency
<<	Coagulation of the solution
<	Cloudy solution
~	Milky solution
>	Transparent solution

Surface activator agents are not only a factor of dispersing particles in the electrolyte but also increase simultaneous deposition. However, there are two problems with the use of conventional surfactant, which are:

- (1) Some of the surfactants remain on the fine particles and enter the coating with them and prevent the deposition of other particles, so in this case the number of deposited particles is limited.
- (2) The adsorbed surfactants affect the morphology of the coating and may have an adverse effect such as brittleness in the coating [36-40].

The effect of a surfactant on simultaneous deposition depends on the following factors:

- (1) Particle dispersion ability.
- (2) Ionic characteristics of surfactants.
- (3) Interaction of electrodes and particles with surfactants.

To create a dispersion stability in the composite plating solution, first, a certain amount of surfactant is dissolved in the solution, then a certain amount of particles is added to the solution and then the solution is stirred for 5 minutes by ultrasonic waves and finally the solution for 30 minutes is mechanically agitated before plating [41-45].

Impact of Plating Current Type

Direct, square, triangular and reverse pulse currents can be used to create a composite coating by electroplating method. Although direct current has been used in many cases to create composite coatings with nanoparticles, studies have shown that the use of pulsed current in addition to the possibility of creating a wider range of composite compounds leads to the presence of more nanometer particles in the coating. Pulse current plating can improve hardness, ductility, surface roughness as well as less porosity and more abrasion resistance. In pulsed current plating, a finer and more uniform structure is available compared to direct current because the growth of crystalline plates is prevented during off times and on the other hand, the nucleation process is encouraged at the beginning of the light time [46-49].

In the pulse current, the pH and the concentration of metal ions on the cathode can be recovered at the off time. The recovery of

metal ions and hydrogen depends to some extent on current density, frequency and duty cycle. The pulse reverse current has the same characteristics as the pc current, except that the cathode current is established during the light time and the anodic current during the off time. This method is known as a successful method for displaying higher concentrations of nanometer particles. Using reverse pulse current, three major advantages can be achieved compared to direct current [50-55].

- (1) More nanoparticles are available in the coating.
- (2) Less concentrations of nanoparticles can be used in solution.
- (3) The selective presence of smaller particles in the coating is achievable.

All three are due to the partial dissolution of the deposited metal during anodic current, so that larger particles tend to separate from the coating while smaller particles remain in the coating.

Impact of Light and Off Times

The results of recent studies on nano composite coatings produced by electroplating method show that the length of off and light times as well as their ratio have a great effect on the crystallographic texture and morphology of the substrate as well as the number and distribution of ceramic particles in the substrate. Increasing the light time in the range of 1 to 5 milliseconds leads to a decrease in grain size, which is due to the increase in potential and, consequently, the increase in nucleation speed. Also, increasing the lighting

time leads to a change in the substrate texture from a random distribution to a strong fibrous texture (200). On the other hand, longer off times cause grain size to increase and crystallographic orientation to change [56-59]. Free ions reach the cathode surface faster compared to ions on the surface of ceramic particles due to their greater mobility. As a result, more free ions are reduced during the light time compared to ions at the particle surface. During off time, because charged particles do not compete with free ions to reach the cathode surface, the particles have a better chance of reaching the cathode surface, which increases poor particles adsorption. As a result, with increasing off time, more particles enter the coating, and this is in agreement with the results of many researchers. However, excessive increase in light time can increase the probability of particles being removed from the cathode surface, which reduces the presence of particles in the coating [60-65].

Impact of Current Density

Since it has been proven today that nanostructured materials have different properties compared to micro-structures, researchers are trying to produce nanostructures and study their properties. In the electrochemical coating method, by controlling the electrochemical parameters, nanostructured coatings can be achieved. One of the important parameters that plays an important role in grain size fineness is the current density. The X-ray scattering pattern obtained from nickel nano crystalline coatings

by applying different current densities. The grain size of nickel nano crystals is obtained from the X-ray scattering pattern (XRD) and using the Scherr discharge relationship. The results are shown in Figure 6: the current density increases to $8 A/dm^2$, the grain size of nickel nano crystals decreases. Since then, the current density has not had a significant effect on the fineness. With increasing current density from 4 to $8 A/dm^2$, the grain size of nickel nano crystalline coatings decreased from 75 nm to 37 nm. The SEM images in Figure 6 also confirm the fineness and uniformity of the coating obtained at higher current densities. The results presented by other researchers are different. Some researchers have reported that an increase in current density in the coating of nickel nanoparticles from the sulfamate bath has resulted in fineness of the grain size, which has been reported as an increase in germination rate. While Cziraki reports that an increase in current density in nickel coating of the sulfate bath has increased the grain size of nickel crystals, what other researchers have found is that in different baths, the effect of current density on nickel nanocrystal fineness is different. At high current densities, with increasing current density, there is no significant increase in fineness, which can be related to two phenomena:

(1) At high currents density, the growth rate of nickel clusters is controlled by mass transfer and hydrogen expansion at the cathode surface.

(2) Deactivation of the cathode surface by the precipitation of nickel hydroxide causes the application of higher current density to increase the passivated surface.

Also, the current density has a great effect on the number of ceramic particles entered in the nano composite coatings produced by electroplating method. In another study on 32 nm alumina particles, it was found that the type of electrolyte affects the presence of ceramic particles in the coating, so that in chloride electrolyte the presence of more nanoparticles in low current density is observed, while in citrate electrolyte in high current density higher concentration of particles were obtained in the coating. This shows that the effect of current density changes with changing electrolyte composition [66-68].

Methods for Determining Dispersed Particles in Coatings

Determination of dispersed particles in composite coatings can be determined by the following methods:

Weighting method

In the weighting method, first the composite coating is dissolved in acid, then the dispersed particles are separated from the solution by filtration and heated. By having the weight of the composite coating and the weight of the dispersed particles, the weight percentage of the particles can be determined.

Microscopic method

This method, also called image analysis, is a common method for determining the volume percentage of particles. The cross-section of the coating is examined with a light or scanning electron microscope and it makes a mistake for particles that are small in size. According to Equation (6), the mass percentage of dispersed particles (G_{dp}) is based on (v_{dp}) where P_{dp} and P_m are the volume mass of the dispersed particles and the ground phase are respectively.

$$G_{dp} = p_{dp} v_{dp} \times 100 / (p_{dp} v_{dp} + p_m v_m) \% \quad (6)$$

v_{dp} , in the opposite relation, is the volumetric concentration of the particle in the coating [69-72].

Electro prop Micro analyzers Method

In this method, the researchers using scanning electron microscope (SEM) micro analyzers, i.e. EDS and WDS determine the elemental concentration of the composite coating, which the percentage of particles in the coating can be calculated by comparing the standard elemental analysis of dispersed particles [73-75].

Photon-related spectroscopy (PCS) method

In this method, the number of scattered particles in the coating is determined using the amount of scanning light reflection of the total particles resulting from the dissolution of the composite coatings in a mixture of sulfuric acid and nitric acid at a temperature of about 40 °C. The PCS method is a simple and fast method with a relative error of less than 2%. This

method gives distinct results even for particles less than 1% by volume, microscopic and micro analytical methods show reproducibility values [76-79].

Conclusion

Abrasion means the loss of material or the removal of material from the surface during which material is removed from one or both surfaces, so abrasion is the result of material removal by physical separation due to brittle break or by chemical decomposition or by melting of materials in the joint of the contact. The presence of mechanical stresses, surface reactions and surface temperature are among the factors causing abrasion in the contact joint. Abrasion mechanisms are divided into four types of adhesion, scratching, fatigue and corrosive, and the abrasion created on a surface may be due to two or more mechanisms. For example, in adhesion abrasion due to friction, particles removed from the surface may be hard work and cause scratches on both surfaces, so both the adhesion and scratching abrasion mechanisms are activated in such a case and cause the material to be removed. Therefore, changes in the abrasion mechanism can be due to changes in surface properties such as surface hardening work, oxide film formation, and surface dynamic reactions due to thermal friction, chemical film formation and abrasion. In adhesion abrasion, the two surfaces are in complete contact with each other and plastic deformation occurs on the surface, in which case the cut occurs at weaker points. There

may be a number of weak points in the harder body, resulting in incisions in the harder body. In scratch abrasion, a hard object enters a soft object and scratches it by deforming the plastic. In adhesion and scratch abrasions, elastic stresses have no effect and in fatigue abrasion, the presence of elastic stresses cyclically causes fatigue abrasion. One of the most important characteristics of metal-ceramic nano composite coatings is their good abrasion resistance, which has led to the widespread use of these coatings in various industries. In general, the abrasion resistance of these coatings on the one hand depends on the toughness, hardness, morphology, grain size, surface roughness and on the other hand on the amount and distribution of the second phase-reinforcing particles. The results show that increasing hardness usually leads to increasing abrasion resistance. Because increasing the hardness, reduces removing of the coating and consequently reduces the abrasion rate. Coating hardness increases with decreasing grain size and increasing the weight percentage of particles and uniform distribution of fine particles. The point to be made here is that increasing the volume percentage of particles does not always lead to an increase in hardness because in addition to the number of particles, the uniformity of their distribution also affects the hardness. In the abrasion test, ceramic particles reduce the abrasion rate by reducing the metal-to-metal adhesive contact. In this way, they increase the abrasion resistance. Clumping of particles in the coating and breaking of clumps on the

surface of abrasion due to scratching abrasion of three bodies increases the abrasion rate of the coating and thus reduces its abrasion resistance. When a second phase hard tooth or particle is pulled hard on a softer surface, it scratches that surface. Scratch abrasion comes in two forms. In the first case, a tooth or the second phase scratches the surface of the body, and in the second case, the scratching or abrasive particles are placed in the space between the two surfaces. The first case is called scratching abrasion of two bodies and the second case is called scratching abrasion of three bodies. Abrasion of three bodies can also be caused by abrasion of two bodies in such a way that the separated particles act as a third particle in the state of two bodies. Since higher surface smoothness leads to reduced involvement of the surfaces in touch. Surfaces with higher surface smoothness have lower abrasion rates.

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