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# Methods for Determining Dispersed Particles in Coatings

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## ABSTRACT

In this research, cream-tungsten carbide nano composite coating was produced by electroplating method. In this section, the equipment used in this research, the materials used along with the test design and the parameters studied in it are mentioned. The highest abrasion rate is obtained at the lowest amount of coating hardness, ie at a frequency of 1 Hz, and the best abrasion resistance at a higher hardness coating, ie at a frequency of 1000 Hz. As can be seen in this diagram, the abrasion resistance of coatings at high frequencies has not improved much despite the presence of particles in these coatings, which can be due to the presence of more agglomerate particles in these coatings. Be. By increasing the frequency from 1 to 1000 Hz, the hardness of the coating increases from HV 590 to HV 705, which is due to the presence of more particles in the coating at high frequencies. Coating hardness increases with increasing presence of particles in the coating and decreases with decreasing presence of particles in the coating.

## GRAPHICAL ABSTRACT



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#### Introduction

Determination of dispersed particles in composite coatings can be determined by the following methods [1-4]. Weighing 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 [5-9]. 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. Electro prop micro analyzers method [10-14].

In this method, using scanning electron microscopy (SEM) micro analyzers, ie EDS and WDS, the elemental concentration of the composite coating is determined, which can be calculated by comparing the standard elemental analysis of dispersed particles with the percentage of particles in the coating. Photon-related spectroscopy (PCS) method [15-17]. In this method, the amount 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, while microscopic and micro analytical methods show reproducibility values [18-21].

#### Abrasion and its mechanisms

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 wear is the result of material removal by physical separation due to brittle failure or by chemical decomposition or by Melting of materials in the joint is the contact. The presence of mechanical stresses, surface reactions and surface temperature are among the causes of wear in the contact joint [22-24]. 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 friction wear due to friction, particles removed from the surface may be hard work and cause scratches on both surfaces, so both the adhesion and scratch wear mechanisms are activated in such a case and cause the material to be removed [25-29]. Therefore, changes in the wear mechanism can be due to changes in surface properties such as surface hardening, oxide film formation, surface dynamic reactions due to thermal friction, chemical film formation and abrasion. In adhesion wear, 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 some weaknesses in the harder body,

and as a result, incisions occur in the harder body. In scratch abrasion, a hard object enters a soft object and scratches it by deforming the plastic [30-34].

In adhesion and scratch abrasions, elastic stresses have no effect and in fatigue abrasion, the presence of elastic stresses but 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 [35-38]. In general, the wear 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 [39-42]. The results show that increasing hardness usually leads to increasing abrasion resistance. Because increasing the hardness, the slowness of the coating and consequently the wear rate decreases. Coating hardness increases with decreasing grain size and increasing particle weight percentage and uniform distribution of fine particles [43-47]. 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 amount of particles, the uniformity of their distribution also affects the hardness [48-51]. 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 [52-55]. Clumping of particles in the coating and breaking of lumps

on the wear surface due to abrasion of the three-part scratch increases the wear rate of the coating and thus reduces its wear resistance. When a second hard tooth or particle is pulled hard on a softer surface, it scratches the 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, scratching or abrasive particles are placed in the space between the two surfaces [56-59]. The first case is called scratching of two bodies and the second case is called scratching of three bodies. Abrasion of three bodies can also be caused by abrasion of two bodies, in which the separated particles act as a third particle in the state of two bodies. Because more surface smoothness reduces the involvement of contact surfaces, surfaces with more surface smoothness have lower wear rates [60-64].

## How to investigate the effect of selected parameters on the microstructure and properties of the coating?

In this study, the effective parameters on plating of cream-carbide tungsten nanostructured composite coating including the concentration of additives and flow density, duty cycle, frequency, concentration of tungsten carbide particles in the cream plating bath were investigated. In this section, the necessary plating conditions of the samples required to investigate the effect of each parameter are described separately, and finally, the effect of evaluating the effect of these parameters will be explained in detail [65-68].

Investigation of the effect diffuser of concentration (SDS)

To investigate the effect of diffuser concentration on the distribution and agglomeration of particles, solutions according to Table 1 were used. The plating temperature was 27 °C and pH=2.5.

<b>Table 1.</b> Plating conditions used to investigate the effect of SDS concentration					
	SDS (g/lit)	Frequency (Hz)	Cycle (%)	Density (A/dm <sup>2</sup> )	
	0-0.5-1-1.5-2	10	50	8	

Investigation of the effect of saccharin additive

To study the effect of saccharin concentration on the distribution, agglomeration and uniformity of the coating, plating was used according to the conditions of Table 2. The plating temperature was 27 °C and pH=2.5.

Table 2. Plating conditions used to investigate the effect of saccharin concentration

SDS (g/lit)	Frequency (Hz)	Cycle (%)	Density (A/dm <sup>2</sup> )
1	10	50	8

## *Investigation of the effect of current density*

To investigate the effect of current density on the volume percentage of precipitated tungsten carbide, plating with different current

densities of 2, 6, 8, 12, 15, 20 amps per square decimeter was performed. The plating temperature was 27 °C and pH=2.5. The plating conditions are given in Table 3 [67-70].

**Table 3.** Electroplating conditions used to investigate the effect of flow density

SDS (g/lit)	Frequency (Hz)	Cycle (%)	Density (A/dm <sup>2</sup> )
1	10	50	2-6-8-10-12-14-16

# Investigation of frequency effect

To investigate the effect of frequency on the amount of deposited tungsten carbide in the coating, four frequencies of 1, 10, 100 and 1000 Hz were applied and it should be noted that the plating temperature is equal to 27 C and pH =

2.5 and the working cycle during these plating is constant. And was equal to 50%. The off and on times at these frequencies are shown in Table 4. Table 5 also shows the plating conditions for the study of these variables.

**Table 4.** On and off times in each frequency

Frequency (Hz)	Shutdown time (msec)	Lighting time (msec)	Cycle (%)
1	500	500	50
10	50	50	50
100	5	5	50
1000	0.5	0.2	50

<b>Table 5.</b> Electroplating conditions used to investigate the effect of frequency					
SDS (g/lit)	Frequency (Hz)	Cycle (%)	Density (A/dm <sup>2</sup> )		
1	1000-100-10-1	50	8		

Investigating the effect of work cycle

The duty cycle changes have been studied as a function of the pulse on and off times in creating the Cr-WC coating. To investigate the effect of the work cycle, the plating temperature was 27 C and pH = 2.5. The plating conditions were in accordance with Table 6.

<b>Table 6.</b> Plating conditions used to study the effect of work cycle
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SDS (g/lit)	Frequency(Hz)	Cycle (%)	Density(A/dm <sup>2</sup> )
1	10	90-70-50-30-10	8

## Conclusion

In this study, the results of the mentioned experiments were presented. First, the plating bath parameters including the concentration of additives and pulsed flow parameters such as flow density, duty cycle and frequency on the particle co-precipitation were studied and then the effect of additives and plating parameters on the hardness and abrasion behavior of the obtained coatings was investigated. As can be seen, with increasing the work cycle from 10% to 90%, the hardness of the composite coating decreases from HV680 to HV610, which is due to the percentage of particles in the coating. Because in work cycle 10, we have the highest percentage of particle. But in the duty cycle of 100, which is the DC current, although the presence of particles does not increase much, but the hardness of the coating increases significantly. In the case of using DC current, one of the reasons that the percentage of particles in the coating increases contrary to the work cycle process is probably due to the presence of agglomerate particles in the coating, which trap and bury the worm's ions in the coating while reducing it. Its remarkable hardness can be seen in the presence of cream hydrides in the coating. In fact, in direct flow, hydrogen gas reacts with cream to produce

cream hard hydrides that have high hardness, and these cream hydrides and the penetration of atomic hydrogen into the coating structure increase the internal stress and hardness of the coating and improve its abrasion resistance. The presence of atomic hydrogen and creamy hydrides in the coating causes a defect in the arrangement and deformation of the crystal structure of the cream. By creating distortion in the network, they produce stress and hardness, which increases the hardness. During the pulse flow, with the continuation of the quenching time, the release of trapped hydrogen gas in the crystal structure of the coating is possible, which in turn reduces the internal stress of the coating, which ultimately leads to a decrease in hardness and prevents cracking of the coating.

## **Conflict of Interest**

The authors declared that they have no conflicts of interest in this work.

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