

*Short Communication*

## **Effect of Particle Size of Tungsten Carbide on Weight Percent of Carbide in Ni-WC Nano-composite**

*Vahid Majazi Dalfard*

Young Researchers Club, Kerman Branch, Islamic Azad University, Kerman, Iran

E-mail: [vmd\\_60@yahoo.co.nz](mailto:vmd_60@yahoo.co.nz)

*Received:* 18 February 2012 / *Accepted:* 11 March 2012 / *Published:* 1 April 2012

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More recently, nickel- tungsten carbide composites are receiving attention as possible wear-resistant coatings for high temperature applications. In this paper, the electrochemical production of Ni-WC nano composite has been investigated by applying direct current (DC). Moreover, three types of carbide with different particle size (purchased, produced by combustion synthesis also known as self-propagating high temperature (SHS) and ball-mill) was used as reinforcement phase. Then composition and morphology of resulted deposit was studied by X-ray diffraction (XRD), scanning electron microscope (SEM) and EDX analysis. The grain size was calculated by Scherrer formula using Xpert High Score software. The evolution of carbide incorporation of Ni-WC nano composite was also investigated by Rietveld analysis of X-ray diffraction using MAUD software. It was found that the amount of embedded WC and thus hardness of composite increases with decrease in particle size of tungsten carbide in the bath. Measurements showed that the grain sizes were between 16-20 nanometers.

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**Keywords:** Electro-deposition, nano composite, nickel, tungsten carbide

### **1. INTRODUCTION**

Nickel-based Composite coatings have more resistance to corrosion than the basic metal has. [1,2] These coatings are used for protection of the automatic parts which are subject to corrosive environments.[3,4] Recently, these coatings have been considered due to resistance to the erosion at high temperature. [5] Nanocrystalline material has high technological importance due to considerable properties such as high hardness, coating resistance, magnetic characteristics and ductility at low temperature in some ceramics. These materials were reported for the first time by Gliter in 1960 [6] and many studies were performed on these materials due to their considerable properties. At present,

many combined techniques have been expanded for production of nanostructured material. These techniques include condensation of gas [6], mechanical milling technique [7], chemical processes[8], sol-gel techniques[9], spray[10], crystallization of amorphous alloys[11] and electric deposition[12-14]] among which electrochemical deposition has been considered due to high purity of the product and lack of major porosity and mass production and short time. In addition, this method is simple, cheap and free from environmental pollution. Geiser and Dehinger reported electrical compound of the rice for the first time in 1932.[15] This method was used gradually as a suitable method for making metals with nanostructure during the recent decade. [16-19] Since tungsten carbide is one of the hard materials which is used in erosion applications,[20] nickel was used as reinforcement phase in this research which can be high property composite.

## 2. MATERIAL AND METHOD

In this research, all materials were purchased from Merck Company and had high purity of 99%. At first, size of carbide particles was measured by Fisher method. For electrical deposit of nanocomposite, suspension was prepared from watts and tungsten carbide particles of which compound is given in table 1.

**Table 1.** suspension compound used for sequestration

raw material	thickness
nickel sulfate	300g/l
nickel chlorine	35g/l
boric acid	40g/l
tungsten carbide	2g/l

PH of the solution was kept 4.7 by adding sulfuric acid and ammoniac solution. In order to mix and increase temperature of the solution, magnetic mixer with speed of 1000 rpm was used and after temperature reached 50 °, WC powder was added with different granulation in 3 different tests. The used carbides include the purchased carbide which has been produced with combustion synthesis method and milled with pellet method for 1 hour. In order to create a uniform suspension, the solution was mixed for 5 minutes with magnetic mixer. In order to create current, antitrust steel (sub-layer) was used with dimensions of 1 cm<sup>2</sup> as cathode and graphite as anode and 2.5 v voltages was applied by rectifier. Current density was considered to be fixed and 0.07A/cm<sup>2</sup>. In order to mix suspension, magnetic mixer was used with cathode manual mixer during deposition to prevent from sedimentation of carbide particles. After 20 minutes, current was disconnected and deposits were tested with XRD and SEM. In order to study size of the grain with Shrer method, Xpert HighScore was used. In order to measure carbide phase, Rietveld analysis was performed on coating diffraction model with MAUD software.

### 3. RESULTS AND DISCUSSION

The calculated sizes for the purchased, synthesized and milled carbides with fisher method include 99, 85 and 39 micrometers.

In diffraction model of figure 1, Ni and WC picks are found well and sharp peak Ni shows field phase and short WC peak shows reinforcement phase as the second phase.

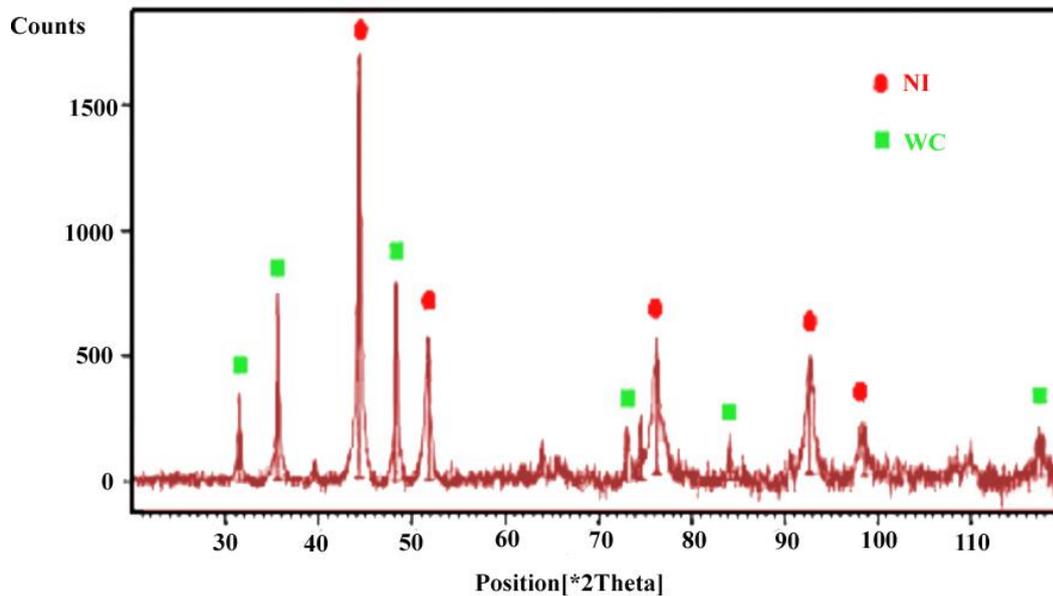


Figure 1. XRD model of nanocomposite coating

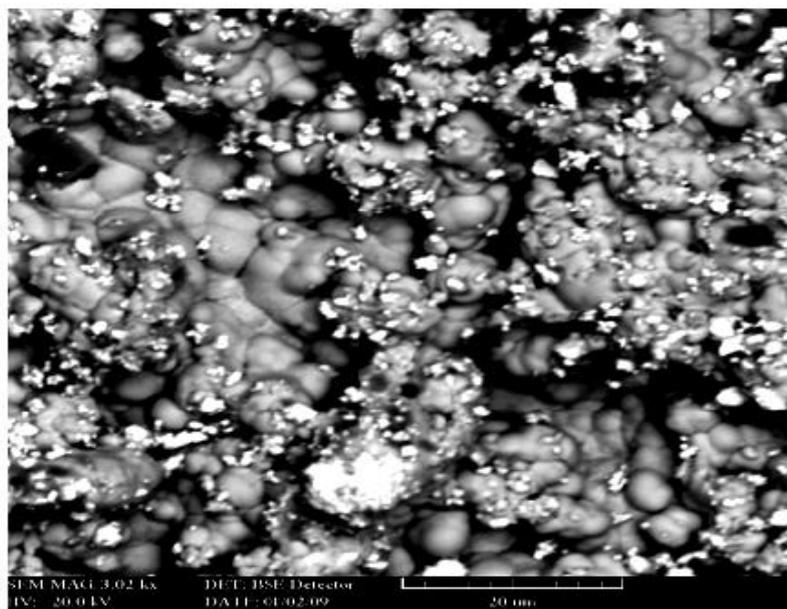


Figure 2. Electron microscope image of the coating produced

SEM image of the obtained coating is given in figure 2 in which two-phased area has been clearly shown. EDX analysis in each phase showed that bright area is tungsten carbide and dark area is nickel which is due to difference of atomic number of tungsten and carbide.

Since the obtained deposit lacks residue stress and the created strain is due to high hardness of the carbide in milled carbide, grain size can be easily calculated which

$$D = \frac{0.9\lambda}{\beta \cdot \cos\theta} \tag{1}$$

$\lambda$  is wavelength in terms of angstrom,  $\theta$  is diffraction angle and  $\beta$  is ray width (in terms of radian) in half intensity of the maximum height. Measurements showed that grain sizes are between 16 and 20 nanometers.

According to the performed researches during electric deposition, hydrogen and oxygen absorption caused to form complexes such as  $NiH^+$  and  $NiOH^+$  and these compounds are included in growth sites and oprevent rom growth of nickel. As a result, nickel field will be created with nanostructure.

Study of phases percentage with Rietveld analysis showed that tungsten carbide amount was more than that of other samples and was about 9% during magnetic mixture with cathode rotation and with use of milled carbide in pellet method. Figure 3 shows Rietveld analysis on coating diffraction model.

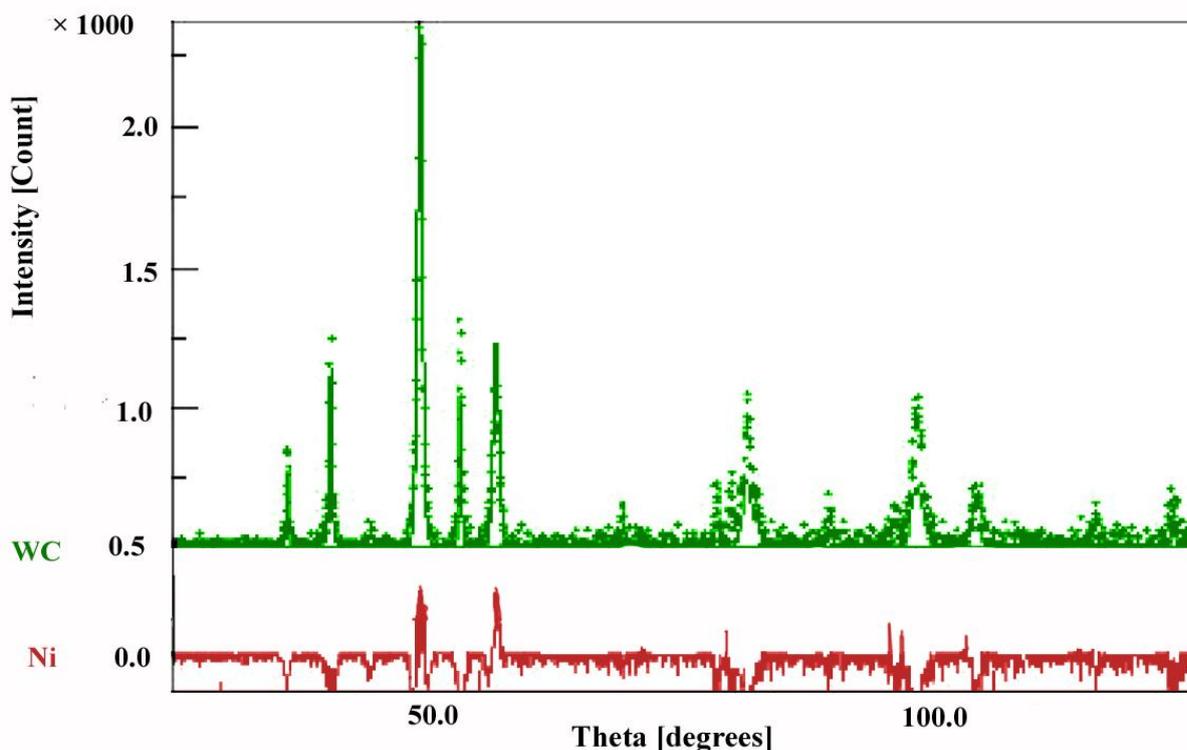
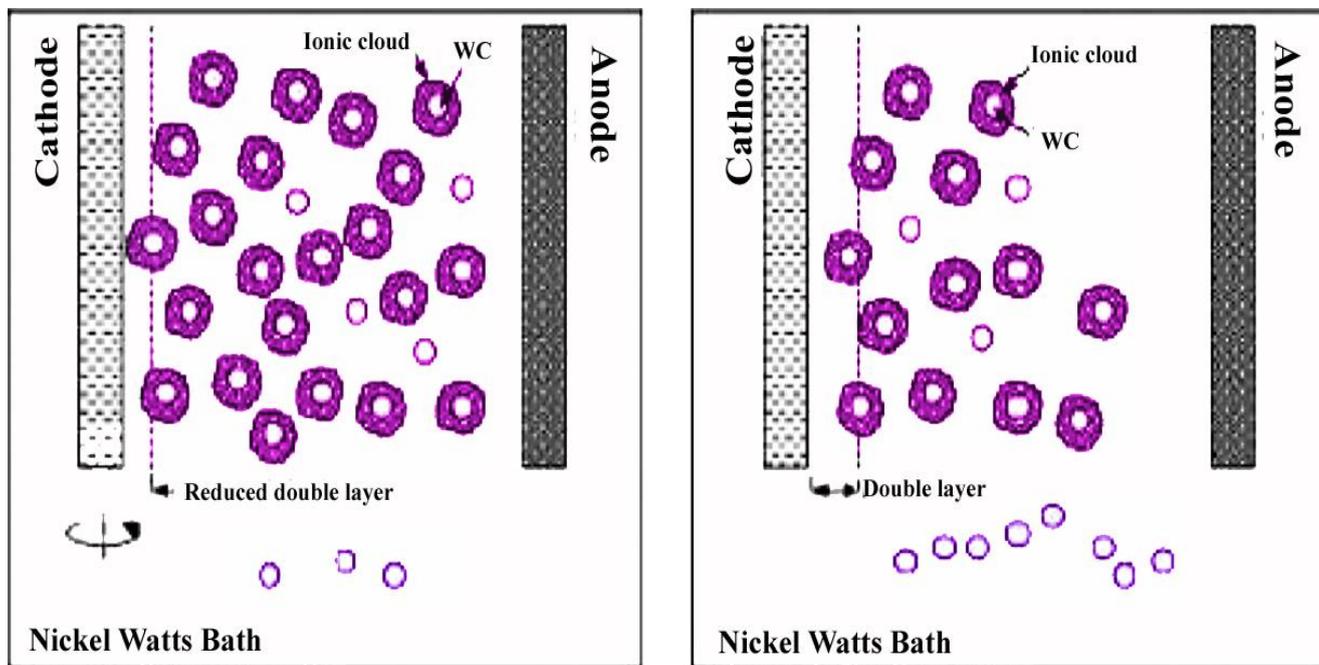


Figure 3. Rietveld analysis on coating diffraction model

Composite deposit mechanism on steel is explained in figure 4. Percentage of carbide involvement and hardness of the coating have increased with cathode rotation in the solution for two reasons:



**Figure 4.** Scheme of nanocomposite production mechanism

Electrolyte uniformity: cathode rotation causes to mix solution well. As result, carbide particles are permitted to float in bath and facilitate absorption of nickel ions around Wc(formation of ionic cloud ) (figure 4-B) and this effect is rarely found in figure 4-A.

Double layer change; thickness of the double layer between electrolyte and sublayer is lower in case of cathode rotation which decreases density of current and increases efficiency of carbide involvement.

#### 4. CONCLUSION

In this research, nickel-tungsten carbide nanocomposite was studied by electrical deposit and effect of carbide granulation on carbide phase percentage was studied. Results showed that the highest percentage of carbide involvement (the highest hardness) relates to the milled carbide which creates uniform distribution of WC in suspension. Phase’s analysis showed that carbide amount as nickel reinforcement phase is 9%. Calculation of grain size with use of Shrer method showed that grains size is between 16 and 20 nm and nanostructure creation mechanism and composite sequestration are given in the report.

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