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## Characterization of size and antiwear ability of nickel and copper nano-particles

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### ABSTRACT

In this paper, the results of a study about using Nickel and Copper nano-particles in a base oil, which is used for engine oil production, are presented. The size characterization and morphology of nano-particles were determined by Atomic Force Microscopy, Electron scanning microscopy and X-ray diffractometry. Dispersion of nano-particles in base oil was achieved by ultrasonic homogenizer and the antiwear ability was evaluated by four ball test. The size determination results of three methods conformed to each other and also with nominal sizes claimed by supplier. The morphologies determined for both nano-particles vouched supplier's claims, too. Copper nano-particles improved antiwear property of base oil but nickel nano-particles did not affect this property.

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### KEYWORDS

Tribology;  
Copper nano particle;  
Nickel nano-particle;  
Base oil;  
Four ball wear tester;  
Atomic force microscopy.

### INTRODUCTION

Nanotechnology is affecting lubrication technology. Adding certain nano-materials to engine oils leads to lower friction and wear of engine parts and consequently higher engine power, lower engine noise and less fuel consumption. Reviewing the literature shows that by using metallic nano-particles in the formulation of lubricants, the friction and wear can be reduced<sup>[1-3]</sup> and it is expected to be more effective in nano scale. In this work, the size and morphology of commercial samples of Nickel and Copper nano-particles were characterized by Atomic Force Microscopy, Electronic scanning microscopy and X-ray diffractometry. Also, the abilities of nano-particles for

enhancing tribological behaviour of base oil were studied by four ball test. Four ball test was chosen because of its relatively lower price, time and sample needed for performing test<sup>[4]</sup>. Because of high surface area and tendency of nano-particles to agglomeration, the dispersion of nano-particles in base oil was achieved by ultrasonic homogenizer<sup>[5]</sup>.

### EXPERIMENTAL

#### Chemicals and instruments

Commercial nano-samples were obtained from Inframat Advanced Materials Company with 80 nanometers nominal diameters. Kinematic viscosities of

base oil at 40 and 100 degree of centigrade were 141.3 and 13.64 centistokes, respectively. Atomic Force Microscope (AFM), Scanning Electron Microscope (SEM), X- Ray Diffractometer (XRD) were Solver P47H of NT- MDT, XL-30 Philips with EDX analysis system and PW- 1840 Philips ( $\text{Cu } k_{\alpha 1}=1.54$ ), respectively. Ultrasonic homogenizer and Four ball test apparatus were UP400S Hielscher and Stanhope-Seta product.

### Atomic force microscopy of nano- particles

Scanning probe microscopy has become a standard technique for obtaining topographical images of surface with atomic resolution<sup>[6,7]</sup>. In addition, they may be used in many applications such as investigation of mechanical, chemical, electrical, magnetic and optical properties of surfaces, study of friction and adhesion forces, modifying a sample surface, crystal growth study and process controlling<sup>[8,9]</sup>. Atomic force microscopy is the most widely used subset of SPM which can be

used in ambient conditions with minimum sample preparation<sup>[10]</sup>. Sample preparation of nano- particles was done according to previous works<sup>[11,12]</sup>.

Sample preparation for nickel nano- particles was done by using ethanol as solvent and Highly Ordered Pyrolytic Graphite (HOPG) as substrate. Nano- particles were dispersed in solvent by applying ultrasonic wave with 35 kHz frequency for 20 minutes at room temperature. Phase and topography images were taken several times in non contact mode with good repeatability. In the case of copper nano- particles same procedure was done except that mica was used as substrate. Sample of 2D and 3D images taken from copper and nickel are shown in Figure 1 and 2, respectively.

### Scanning electron microscopy of nano- particles

Nano- particles were dispersed in ethanol and SEM images were taken. Nano- particles diameter range and size distribution are shown in TABLE 1.

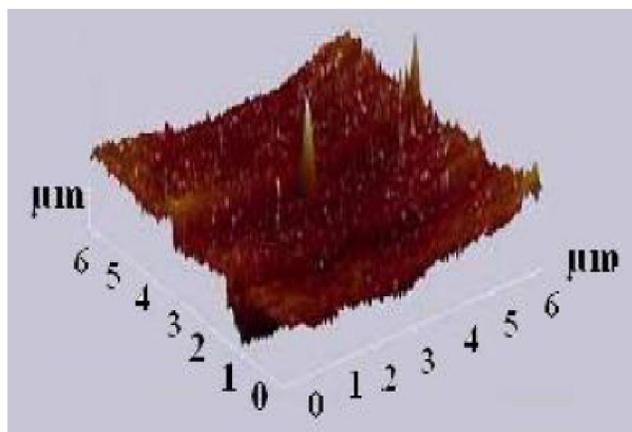
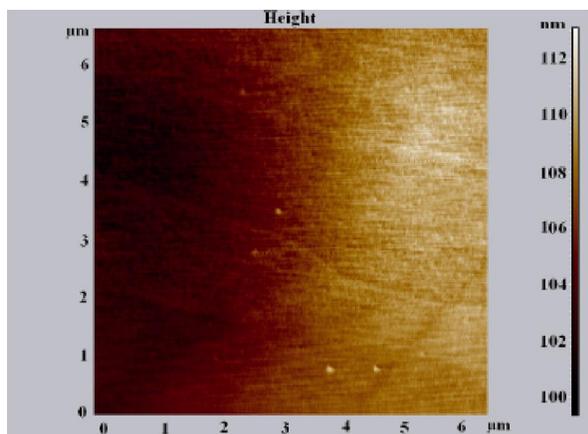


Figure 1 : 2D and 3D AFM images of nickel nano- particles

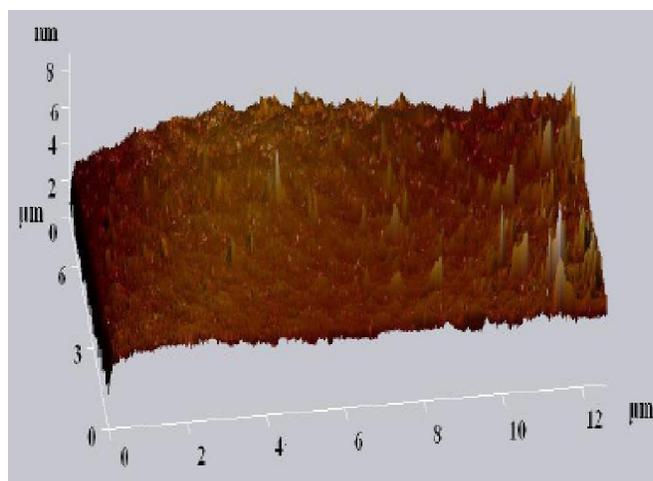
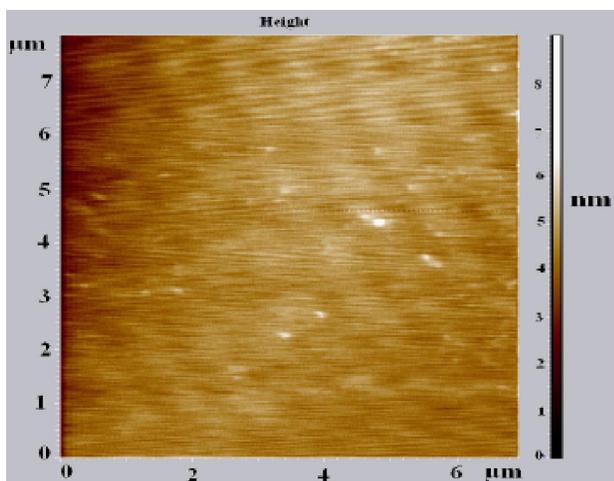


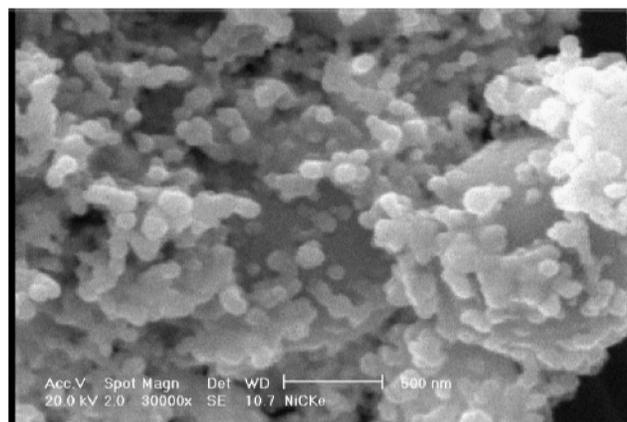
Figure 2 : 2D and 3D AFM images of copper nano- particles

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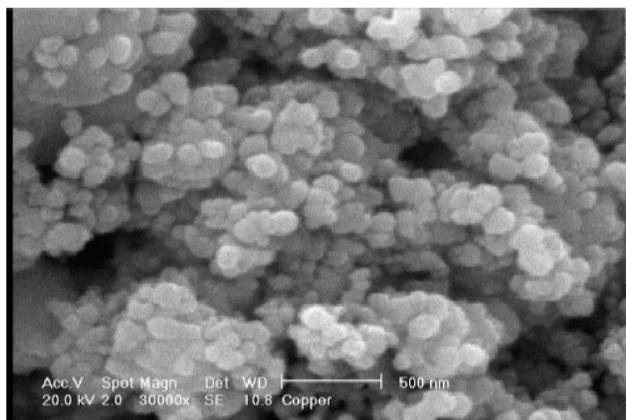
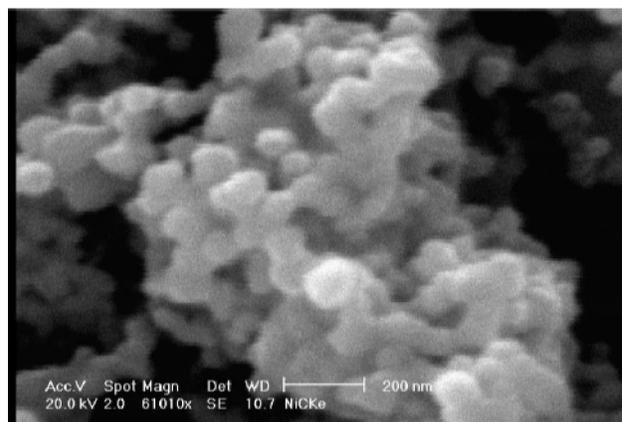
SEM images of nickel and copper nano-particles are shown in Figure 3 and 4, respectively.

**TABLE 1 : The size distribution of nickel and copper nano-particles according to SEM results**

Diameter range (nm)	Distribution (%)	
	Nickel	Copper
0-50	17	23
50-100	58	61
100-150	20	15
150-200	5	1



**Figure 3 : SEM image of nickel nano-particles**



**Figure 4 : SEM image of copper nano-particles**

copper (JCPDS 4-836). There are some CuO and Cu<sub>2</sub>O impurities in the copper sample which can be related to high activity of copper nano particles to oxidation. The size of nickel and copper nano-particles, determined by Scherer method, were 70 and 85 nm, respectively. Both phases have cubic structure.

### Comparison of AFM, SEM and XRD results

The size determination results of nano-particles with AFM, SEM and XRD methods are shown in TABLE

### X-ray diffractometry of nano-particles

Powder X-ray Diffraction is one of the primary techniques used by mineralogists and solid state chemists to examine the physico-chemical make-up of unknown materials. XRD is an easy tool to determine the size and the shape of the unit cell for any compound<sup>[13]</sup>. XRD pattern of nickel and copper nano-particles are shown in Figure 5 and 6, respectively. In Figure 5, 44.507 degree peak is related to nickel (JCPDS 4-850). In Figure 6, 43.297 degree peak is related to

2. It is shown that the size determination results conformed to each other and also to nominal size. The morphologies determined for both nano-particles vouched supplier's claims, too.

### Tribological behaviour of nano-particles

Nano-materials tend to be agglomerated when being mixed into a liquid. Therefore effective means of deagglomerating and dispersing are required to overcome the bonding forces after wetting the micron-

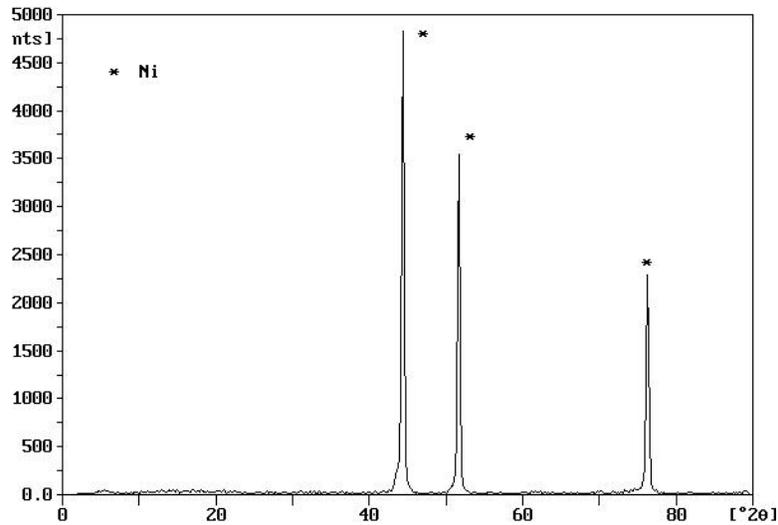


Figure 5 : XRD pattern of nickel nano- particles

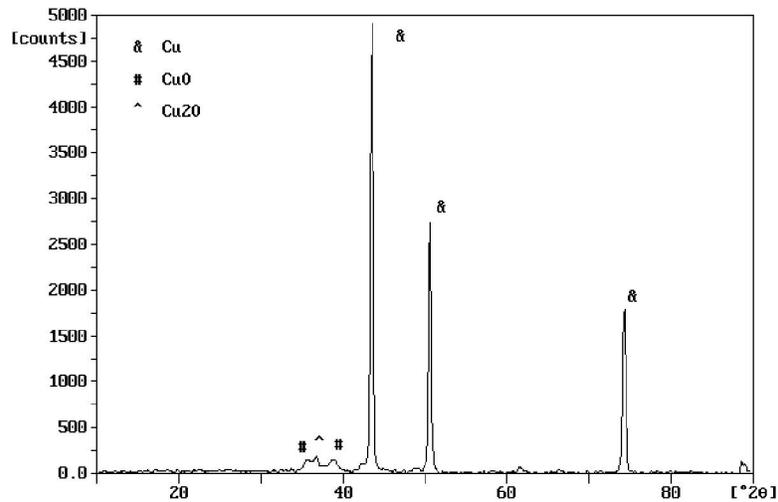


Figure 6 : XRD pattern of copper nano- particles

TABLE 2 : The results of size determination of nano-particles with AFM, SEM and XRD methods

Nano-particles	Nominal	Mean diameter (nm)				XRD	
		AFM	SEM				
Nickel	80	84	0-50	50-100	100-150	>150	85
			(17%)	(58%)	(20%)	(5%)	
Copper	80	77.6	0-50	50-100	100-150	>150	70
			(23%)	(61%)	(15%)	(1%)	

powder or nano-powder. For this application, ultrasound has proven to be more effective than many other devices<sup>[14]</sup>. In this work nano-particles were dispersed in base oil by ultrasonic homogenizer at 240 W power for 30 minutes as one pulse per second wave applying. The dispersing vessel was cooled in order to prevent overheating resulted from applying. Reviewing the literature shows that by using metallic nano-particles in the formulation of lubricants, the friction and wear can

be reduced. The results of four ball tests according to ASTM D 2783 are shown in TABLE 3.

As shown in TABLE 3, addition of nickel nano-particles to base oil did not improve wear diameter and the welding load of base oil. Increasing the amount of nano-particles from 0.2 to 1.0 weight percent did not change the situation, too. Addition of copper nano-particles to base oil reduces wear diameter and improve the welding load. Increasing the amount of copper nano-particles from 0.2 to 1.0 weight percent enhances the effect.

**Viscosity behaviour of dispersion of nano-particles in base oil**

Kinematic viscosities of base oil without and with 0.2 and 1.0 weight percent nano-particles are shown in TABLE 4.

As shown in TABLE 4, adding nano particles to base

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**TABLE 3 : The results of four ball tests according to ASTM D 2783**

Load (kg)	Base oil	Wear diameter (mm)			
		Base oil+ nano nickel		Base oil+ nano copper	
		0.2 wt%	1.0 w%	0.2 wt%	1.0 w %
100	2.327	2.180	2.127	2.157	1.967
112	2.383	2.473	2.240	2.217	2.183
126	2.393	2.673	2.417	2.383	2.363
141	2.760	2.703	2.593	2.567	2.483
158	2.873	2.977	2.893	2.777	2.567
178	welding	welding	welding	2.893	2.693
200				welding	2.940
224					3.307
251					welding

**TABLE 4 : Kinematic viscosities of base oil with and without nano- particles**

Lubricant	Kinematic viscosity at 40° C	Kinematic viscosity at 100° C	Viscosity index
Base oil	141.3	13.64	91
Base oil+ 0.2 % nano nickel	140.2	13.58	91.13
Base oil+ 1.0 % nano nickel	140.1	13.57	91.09
Base oil+ 0.2 % nano copper	141.2	13.63	90.95
Base oil+ 1.0 % nano copper	140.4	13.61	91.35

oil does not change significantly the viscosity behaviour.

## CONCLUSION

The morphology and size of commercial samples of nickel and copper nano- particles were determined by Atomic Force Microscopy, Electron scanning microscopy and X- ray diffractometry. The size determination results agree with each other and also with nominal sizes. The morphologies determined for both nano-particles vouched supplier's claims, too.

Addition of nickel nano- particles to base oil did not enhance the wear property. Increasing the amount of nano- particles from 0.2 to 1.0 weight percent did not change the situation, too.

Addition of copper nano- particles to base oil improved antiwear property and increasing the amount of copper nano- particles from 0.2 to 1.0 weight percent enhanced the effect.

Adding both nano- particles to base oil does not change significantly the viscosity behaviour.

## ACKNOWLEDGEMENTS

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