



# Tribological behaviours of lubricating oils with CNT and Si<sub>3</sub>N<sub>4</sub> nanoparticle additives

**M.N. Çöl, O.N. Çelik, A. Sert\***

Department of Mechanical Engineering, Eskişehir Osmangazi University,  
26480 Eskişehir, Turkey

\* Corresponding e-mail address: asert@ogu.edu.tr

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## ABSTRACT

**Purpose:** The goal of this work is comparative study of lubricants with different content of nanoparticle additive to obtain material with an optimal tribological properties.

**Design/methodology/approach:** The effects of carbon nanotube (CNT) and Si<sub>3</sub>N<sub>4</sub> nanoparticle additives on the lubricated friction and wear behaviour of AISI 4140 steel were investigated. In tribological tests, the amounts of nanoparticles added to base oil (SAE 10W40) were varied. The ball-on-disc configuration of the tribometer was used for wear testing (DIN 50324). Specific wear rate, coefficient of friction, wear scar depth were measured. The wear surfaces were analysed using scanning electron microscopy and surface profilometer.

**Findings:** Results showed that with the addition of nanoparticles, wear rates of AISI 4140 steel were decreased. The best additive content of synthetic oil was 0.05% CNT + 0.05% Si<sub>3</sub>N<sub>4</sub> (mass fraction), and in this case, the friction coefficient and the wear rate were decreased 15.2% and 76.4%, respectively.

**Research limitations/implications:** Obtained lubricants are characterized by tribological and frictional properties but homogeneity of lubricants should be improved because of agglomeration. Farther works will be continued also for different contents of lubricant.

**Practical implications:** Tested lubricants can be applied among in industry where wear and friction properties are important.

**Originality/value:** Nanoparticle additive oils which have excellent tribological properties can be very attractive for automobile industry. New type of lubricant mixture (CNT and Si<sub>3</sub>N<sub>4</sub>) is used in the paper.

**Keywords:** Wear resistance; Lubricant; Nanoparticle; CNT; Si<sub>3</sub>N<sub>4</sub>

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## PROPERTIES

## 1. Introduction

Recent investigations of nanotribology have revealed that nanoparticles have excellent properties for lubricant-based tribological applications, such as high load capacity, wear resistance and friction reduction [1]. In the case that the concentration of nanoparticles is less than 2%, the tribological properties of the base oil with nanoparticles exhibit good friction and wear properties according to the settlement between the rubbing surfaces [2].

The friction-reduction and anti-wear properties of oil with nanoparticle additives are based on: (a) the nearly spherical geometry of nanoparticles, which causes them to act as a rolling medium when the machine parts move, are expressed as a rolling effect, (b) the nanoparticles embedded in the surface asperities and cracks create a mending effect, (c) the protective film containing nanoparticles and the base oil create a thin layer between the frictional surfaces to reduce friction and wear, and (d) hard nanoparticles may act as polishers by eliminating asperities on surface roughness [3].

On previous research of different types of nanoparticles are investigated, such as metal [4], oxide [5], sulphide [6], borate [7], carbonate [8], carbon material [9], polymers [10] and rare-earth compounds [11].

The aim of this study is to examine the tribological properties of lubricating oils with and without  $\text{Si}_3\text{N}_4$  and CNT nanoparticles by characterizing the worn surfaces with SEM and surface profilometer.

## 2. Material and method

In this study, the tribological effects of the nanoparticles  $\text{Si}_3\text{N}_4$  and CNT are measured by adding into the engine oil both solitary and mixed at definite proportions. Being reference experiment, the abrasion of the steel AISI 4140 in boundary lubrication condition is taken into consideration in base oil environment and it is compared with the experiments which were done with the addition of the nanoparticles.

### 2.1. Materials

In this study, with the purpose of analysing the tribological properties, the nano additions used in the oil are  $\text{Si}_3\text{N}_4$  and CNT.  $\text{Si}_3\text{N}_4$  is a ceramic material which has high resistance at wide temperature range, low thermal expansion coefficient and high modulus of elasticity. It has

the fracture toughness at the level of which is considered high for ceramic materials. The  $\text{Si}_3\text{N}_4$  nanoparticles used in experiments are provided from the firm Alfa-Aesar and the degree of purity is 98.5%.  $\text{Si}_3\text{N}_4$  nanoparticles are amorphous structure and nano sized. The detailed information about  $\text{Si}_3\text{N}_4$  nanoparticles is given in Table 1.

Table 1.  
The properties of materials

Nanoparticles	Properties:
CNT	Morphology: multiwall, purity: 95%, particle size: 3-20 nm, density: 1.31 g/cm <sup>3</sup>
$\text{Si}_3\text{N}_4$	Morphology: nearly spherical, purity: 98.5%, particle size: 15-30 nm, density: 3.4 g/cm <sup>3</sup>
Base oil	Physical properties:
SAE10W40	Density (15°C): 0.871 g/cm <sup>3</sup> , Viscosity index: 871 Viscosity: 40°C (ASTM D445) – 90.8 cSt 100°C (ASTM D445) – 14.1 cSt
Specimens	Properties
Substrates	AISI 4140 steel, hardness: 52.1 HRC, $R_a=0.02$ , dimensions: $\phi 30 \times 10$ mm
Balls	WC-Co (6%) cemented carbur, hardness: 91.6 HRC, E: 690 GPa, d: 3 mm

The other additive oil material CNT has the modulus of elasticity and tensile strength. It can stay stabilized up to 3000°C temperature. The CNT addition used in experiments is Alfa-Aesar product and the degree of purity is 95%. The CNT nanoparticles are averagely 3-20 nm and have the feature of “multiwall”. The detailed information about CNT nanoparticles is given in Table 2.

Table 2.  
Experiment plan

Sample Number	$\text{Si}_3\text{N}_4$	CNT	Sample Number	$\text{Si}_3\text{N}_4$	CNT
1	0	0	7	0	0.1
2	0.1	0	8	0	0.2
3	0.2	0	9	0.05	0.05
4	0.5	0	10	0.1	0.1
5	0.8	0	11	0.13	0.067
6	1	0	12	0.067	0.13

The synthetic oil SAE 10W40 was used with the purpose of analysing the tribological effects of the steel AISI 4140. In the oil which was used, the tribological properties were analysed with definite proportions of nanoparticle additions. The added nanoparticles were mechanically mixed in the way to enable the particles to diffuse homogeneously at the room temperature. The properties of the base oil are given in the Table 1, the mixing ratios of oil and nanoparticles are given in the Table 2. Among the experiments, there is also oil environment without particle addition.

The steel AISI 4140 (composition: 0.38-0.43% C, 0.75-1% Mn, 0.15-0.3% Si, 0.8-1.1% Cr, 0.15-0.25% Mo) which is commonly used in automotive industry was used as base material. Before the tribological tests, the steel AISI 4140 was quenched and then tempered. As a result of thermal processing, the structure of the material's metallographic analyses is seen as martensitic structure (Fig. 1). Additionally, all base materials were polished before the tribological tests and their surface were cleaned. The properties of the steel AISI 4140 are given in Table 1.

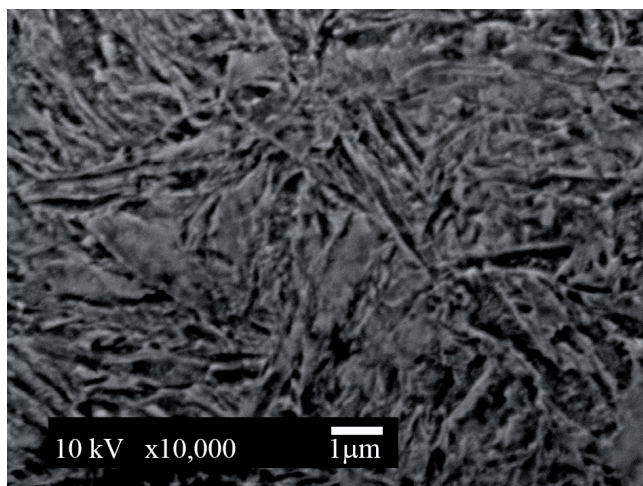


Fig. 1. Microstructure of quenched AISI 4140

## 2.2. Tribological tests

Tribology tests were done in CSM Tribometer device and at the standard of DIN 50324. Experiment geometry is in the shape of ball-on-disk. In the experiments, WC-Co (6%) balls were used as opposite object to the steel AISI 4140. Their globosity and compound are certificated, the balls were supplied from the firm Redhill Precision. The properties of the balls used are given in the Table 1. In the tribology tests, the abrasion and friction properties of

the steel AISI 4140 were derived in different lubricant environment. The parameters used in tribology tests are given in Table 3.

Table 3.

Tribological test operating condition

Ball	ø3 mm, WC-Co (6%)
Sliding specimen	Quenched AISI 4140
Applied load	10 N
Type of motion	Rotating
Sliding velocity	5 cm/s
Sliding distance	40 m
Temperature	Ambient
Lubricant	Base oil, modified base oil with additives

Before tribology tests, the surface of the steel AISI 4140 were polished and cleaned with ethanol. Then, samples' surface roughness value was measured with the device Mitutoyo SJ-400. After the tests, the same device was used with the purpose of measuring the worn surface profiles. After the tests, coefficient of frictions which are dependent to different lubricant environments were derived from CSM Tribometer device. To be able to have the samples' worn surface profiles JEOL-JSM 5600LV scanning electron microscope (SEM) was used.

## 3. Results and discussion

### 3.1. Friction reduction properties

The frictional properties of the steel AISI 4140 were analysed with rotating tribotester under the 10 N load. Figure 2 shows the coefficients of friction of the base oil with and without nanoparticles. When the Figure 2 is analysed, after the tribological tests which were done with nanoparticle added oil, it is seen that frictional properties get better in terms of base oil except one situation. The results change with respect to the addition degree of the nanoparticle and kind of it. This change can be said to be dependent to the nanoparticles' settlement to the contact surface and their being too small in those settlements than the thickness caused by the oil. With this way mutual contact is avoided; friction and wear properties get better. However, this situation can be reversed by increase of the nanoparticle concentration. Wear and friction properties can deteriorate [5,12]. Generally, the nanoparticle addition into the oil repairs by filling the contact surfaces of those particles and prevents the

contact by creating boundary layer lubricating and decrease the coefficient of friction [13,14].

### 3.2. Anti-wear properties

The wear properties of the steel AISI 4140 are specified according to the specific worn rate both with base oil and nanoparticle added oil after the worn distance of 40 m. All the specific worn rates in the experiment conditions are given in the Figure 3. When the values are analysed, the specific worn rates are lower in the nanoparticle added oil compared to the base oil. The recovery values of the wear properties with nanoparticle addition are between 15.2 and 76.4%. In Figure 3, one group of different ratios of  $\text{Si}_3\text{N}_4$  added, one group of different ratios of CNT added and the

other group of different ratios of  $\text{Si}_3\text{N}_4$ -CNT added situations are shown. When the  $\text{Si}_3\text{N}_4$  added situation is analysed, it shows better wear results compared to the base oil. It shows different properties with the different addition ratio. When CNT added two situations are analysed, a better wear result is seen compared to the base oil and specific worn rate increases dependent to the addition ratio's increase. When the different addition ratios of  $\text{Si}_3\text{N}_4$  and CNT are analysed, it can be said that specific worn rate increases with the CNT increase. As a result of a research done with MWCNT, it is indicated that the wear and friction properties are dependent to the diffusion of the nanoparticles in the oil [15]. When all then values are analysed, it can be said that nano additions have a mending effect on the wear and the tests done with the mixture of  $\text{Si}_3\text{N}_4$  and CNT are more stable.

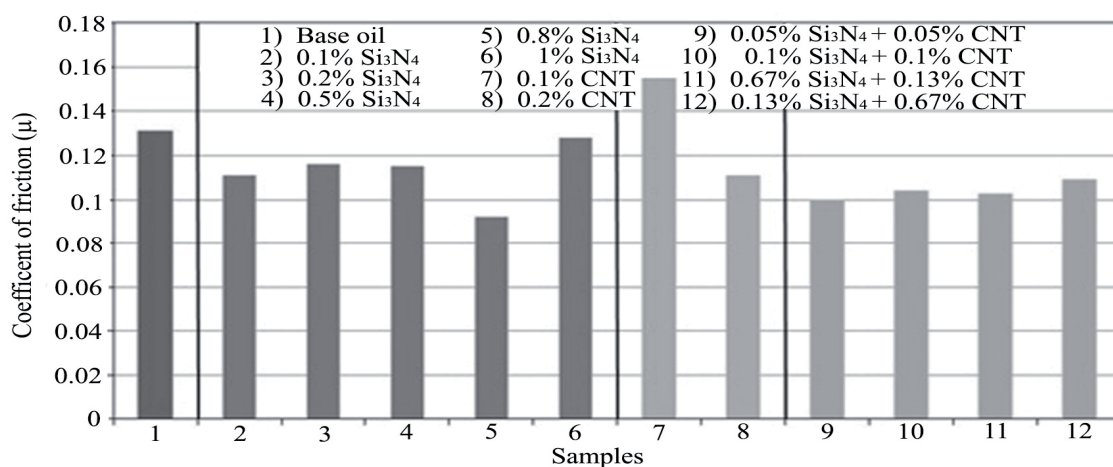


Fig. 2. The average coefficient of friction of base oil and nanoparticle added oil

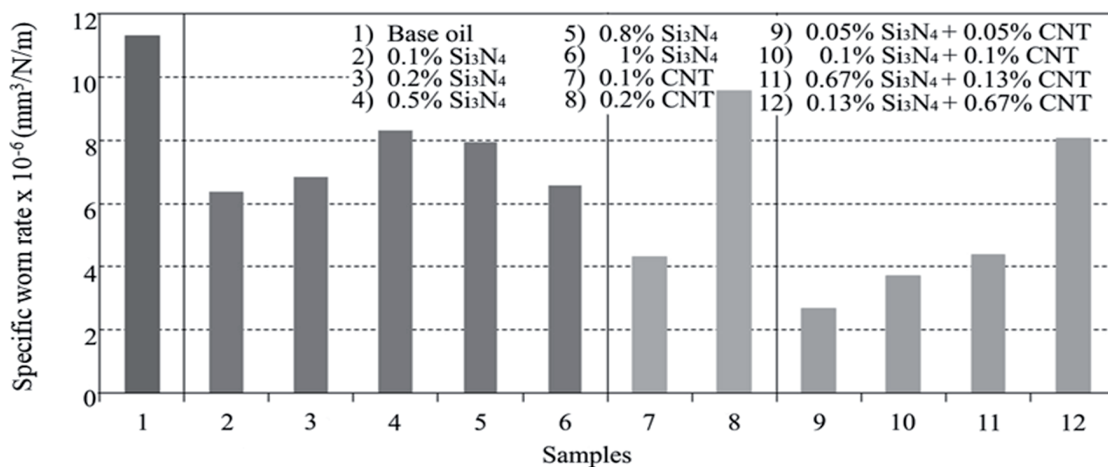


Fig. 3. The specific wear rates of base oil and nanoparticle added oil

In the nanoparticle added lubricants, it is indicated that the particles in the oil can have agglomeration and precipitation with the increase of particle concentration and lubricant properties decrease in such a situation [16]. It is possible that more particles hold on to the contact surfaces and boundary lubricating film gets thicker and consolidates with this way. With this film, the contact with the asperities at the contact surface is prevented and wear and friction values decrease. With more incrementation of particle concentration, coagulations happen between the friction surfaces and this transforms the friction unstable. As a result, high friction and wear values can be seen [17].

Worn scar depth was measured so as to measure the specific worn rates. In Figure 4, base oil and nanoparticle added situations are seen. In Figure 4a, 0.05% Si<sub>3</sub>N<sub>4</sub>+0.05% CNT nanoparticle added lubricant's worn scar depths showing the least abrasion with base oil are seen. It is seen that the abrasion deepness is more intense under 10 N load without nanoparticle when compared to

nanoparticle added situations. In this case, it can be said that nanoparticles create a conveyor effect between the substrate and the ball [3]. Additionally, lower worn scar depth can be associated with the nanoparticles' rolling effect. Nanoparticles can decrease shearing stress by accumulating on the abraded surface and decreases abrasion [18]. In the previous studies, it was indicated that added nanoparticles shows a diminishing effect on wear by slipping over each other of mutual two surfaces with rolling effect, by filling the asperities on the contact surfaces as a result of the wear with mending effect [12].

In Figure 4b, 0.2% CNT added oil's worn scar depth showing the most abrasion with base oil is seen. As it is seen in Figure 3, 0.2% CNT added oil's worn scar depth ratio is the highest value after the base oil. Depending on this, their worn scar depths are close to each other as their specific worn rates. This situation is being agglomerated with the increase of the CNT amount and effecting the wear badly [16].

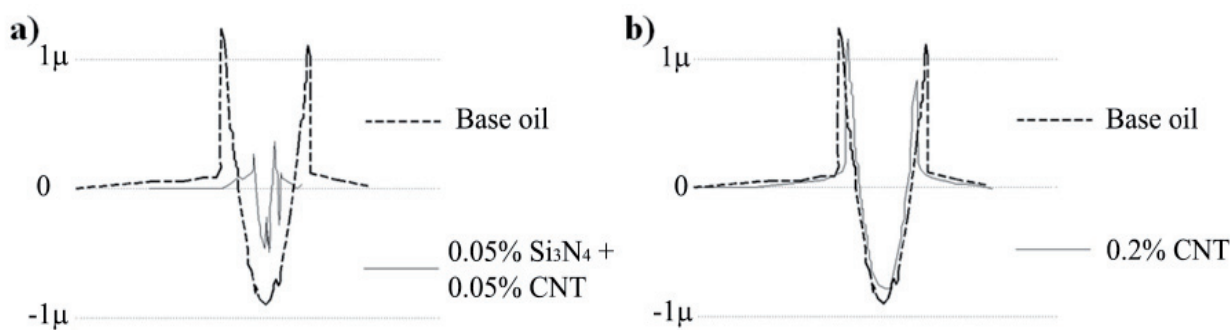


Fig.4. Comparison of worn scar depth, a) base oil with 0.05% Si<sub>3</sub>N<sub>4</sub>+0.05 CNT, b) base oil with 0.2% CNT

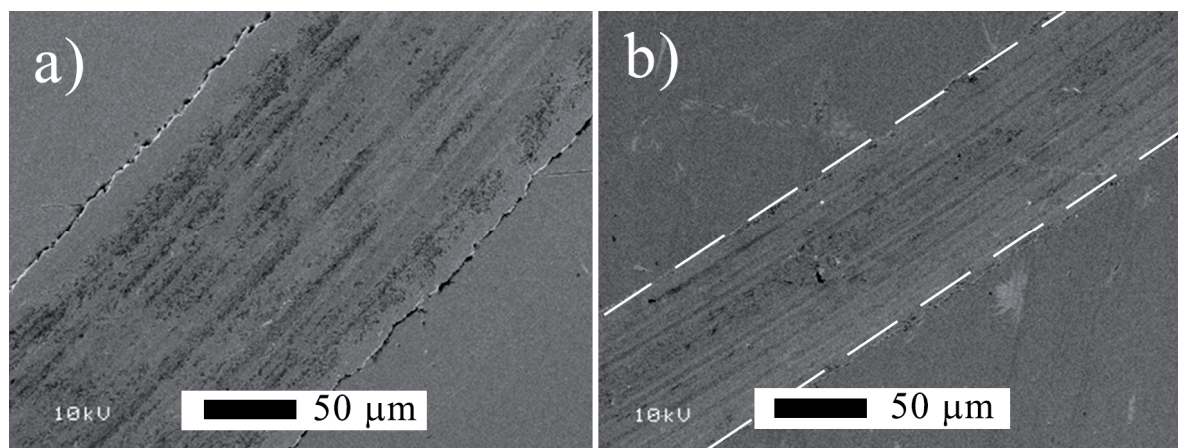


Fig. 5. SEM morphology of worn surfaces of AISI 4140, a) base oil and b) the oil containing 0.05% Si<sub>3</sub>N<sub>4</sub>+0.05% CNT

After the wear test experiments done in Figure 5, SEM images which show the profiles of the worn areas were given. Figure 5a belongs to the base oil and the Figure 5b belongs to the 0.05% Si<sub>3</sub>N<sub>4</sub>+ 0.05% CNT added oil having the least abrasion. In Figure 5a, grooves which are in the direction of sliding, shallow and parallel to each other are seen on the surface as a result of the wear test with base oil used. In this case, worn profile width is measured as 164.6 µm. In Figure 5b, grooves which are in the direction of sliding, shallow are seen. With the nanoparticle addition into the oil, decrease is seen in the profile width and profile width is measured as 90 µm. After all the other tests, the worn profile widths are measured and seen in direct proportion with specific worn rates.

#### 4. Conclusions

As a result of the tribological tests done with nanoparticle added and base oil;

In both two different experiments with nanoparticle addition, it is determined that nanoparticles have a repairing effect on wear. Generally, it can be said that frictional properties get better with nanoparticle addition.

As a result of the test done with the best condition to abrasion – 0.05% Si<sub>3</sub>N<sub>4</sub>+0.05% CNT added oil – 76.4% recovery was seen.

It is seen that friction and abrasion properties change in terms of the amount of the additives Si<sub>3</sub>N<sub>4</sub> and CNT.

When the ratios of individual added particle and two different particles are compared, the binary situation is seen as more successful.

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#### Additional information

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