

**TRIBOLOGICAL BEHAVIOR OF IF-WS₂ NANOPARTICLE ADDITIVES IN
SIMULATED ENGINE CONDITIONS**

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

**IF-WS₂ NANOPARÇACIKLARIN SİMÜLE EDİLMİŞ MOTOR
ŞARTLARINDA TRİBOLOJİK DAVRANIŞLARI**

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ABBREVIATIONS

TDC	: Top Dead Center
BDC	: Bottom Dead Center
IF	: Inorganic Fullerene-like
BSFC	: Brake Specific Fuel Consumption
FMEP	: Friction Mean Effective Pressure
rpm	: Revolution per Minute
CA	: Crank Angle
BMEP	: Brake Mean Effective Pressure

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TRIBOLOGICAL BEHAVIOR OF IF-WS₂ NANOPARTICLES IN SIMULATED ENGINE CONDITIONS

SUMMARY

This study aims to investigate tribological behavior of Inorganic Fullerene like WS₂ nanoparticles added into mineral oil. A special purpose cylinder liner- piston ring friction simulator is used for experimental analysis. Different concentrations of IF-WS₂ nanoparticles are prepared to see the effects of the concentration on tribological performance. Labview software is used for data acquisition and MATLAB scripts are used for post processing. The effects of running in under a specified load and speed for specified time is also applied to observe a tribofilm potentially formed on the contact surfaces. A test matrix is used, and the tests are done under both hydrodynamic and mixed lubrication regimes. The experiments showed more than 30% of reduction in friction coefficient with the use of 10 % IF-WS₂ concentrated engine oil. In cold run, for hydrodynamic lubrication, no effect on friction reduction is observed. The experiments are repeated with reference oil to see the residual effects of nanoparticles additives. Although friction coefficient is raised somewhat with the use of reference oil, it is still found to be less than the first results. Raman spectra analysis is used to investigate the composition of the liner surface after the experiments and the results are shown within the graphs.

As a result, frictional behaviors of standard and IF-WS₂ nanoparticle formulated engine oils are compared using a specific liner-ring friction simulator. Practical use of nanoparticle additives in engine oils are discussed.

IF-WS₂ NANOPARÇACIKLARIN SİMÜLE EDİLMİŞ MOTOR ŞARTLARINDA TRIBOLOJİK DAVRANIŞLARI

ÖZET

Bu çalışma, madeni motor yağı içerisine ilave edilmiş olan soğanımsı yapıdaki WS₂ nano parçacıkların tribolojik performansının deneysel olarak incelenmesini hedeflemektedir. Deneysel analiz için özel amaçlı bir silindir duvarı – piston segmanı sürtünme simülatörü kullanılmıştır. Konsantrasyonun etkisini gözlemek için değişik konsantrasyonlarda nanoparçacık içeren motor yağı karışımları hazırlanmıştır. Deney verilerin toplaması için NI LabView yazılımı kullanılmış olup, test sonrası incelemeler için ise MATLAB kodları hazırlanmıştır. Yüzeyde oluşmuş olası tribofilmin gözlemlenmesi için, belirli bir yük ve hız altında ve belirli bir süre alıştırma deneyleri yapılmıştır. Bir test matrisi kullanılmış olup, deneyler hidrodinamik ve karma yağlama rejimlerinde yapılmıştır. Deneylerde, % 10 WS₂ nanoparçacık içeren yağın kullanımıyla sürtünme katsayısında % 30'dan fazla bir düşüş gözlenmiştir. Soğuk çalışmada ise, hidrodinamik yağlama rejimi için sürtünme katsayısında herhangi bir değişiklik gözlenmemiştir. Nanoparçacıkların yüzeyde kalıcı etkisini görmek için deneyler referans yağ ile tekrarlanmıştır. Referans yağın kullanımıyla sürtünme katsayıları bir miktar yükselmiş olsa da, hala ilk referans yağ sonuçlarından daha düşük oldukları bulunmuştur. Sürtünmedeki kalıcı düşüşe neden olan yüzey oluşumunun incelenmesi için yüzey Raman spektroskopisi tekniği ile incelenmiş, ve sonuçları grafikler içerisinde gösterilmiştir.

Sonuç olarak; özel bir silindir duvarı–piston segmanı sürtünme simülatöründe, standart mineral yağ ile IF-WS₂ nanoparçacık içeren yağların tribolojik davranışı karşılaştırılmıştır. Motorlarda nanoparçacık içeren yağların pratikte kullanımı tartışılmıştır.

1 INTRODUCTION

Environmental concerns late in the 20th century resulted strong regulations for automotive manufacturers to develop more fuel efficient, compact automobile engines with nearly zero air pollutant emissions. More compact engines imply higher temperatures and pressures inside the combustion chamber for higher thermal efficiency. This result in higher loads on moving contact surfaces and lower viscosity of oil due to rise in temperature; however excessive lubrication which causes oil consumption is limited by regulations [1].

The major power loss of an IC engine is the thermal losses, which are limited by the laws of thermodynamic. The other losses are the mechanical losses due to friction of contact surfaces. The mechanical losses can be grouped as the drive train losses and the engine losses. Engine losses consist of valve train and auxiliary losses but the power cylinder losses take important place in engine friction. Figure 1.1 shows the percentage of piston group power losses, also called power cylinder losses [2].

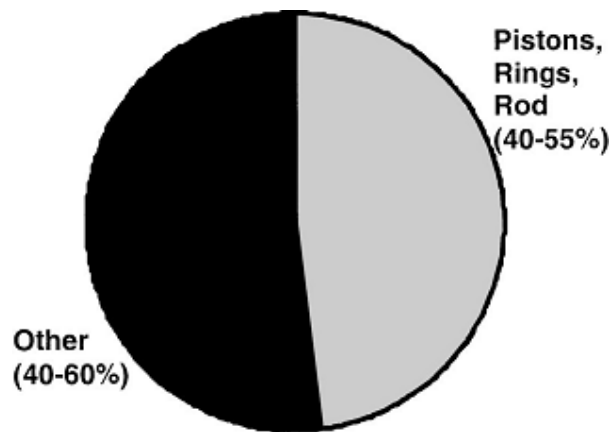


Figure 1.1: Distribution of total engine mechanical friction [2]

1.1 Background

Piston rings are the sealing elements for gas flow from combustion chamber, and control the oil film thickness on cylinder liner to limit oil consumption. In earlier

times, there were not any rings used in a steam engine design due to lower temperatures and pressures. In consequence of high power demand, today's IC engines have higher temperatures and hence higher thermal loads on the piston and the liner. The clearance of the piston and the liner changes due to temperature rise, therefore the rings are designed to limit the gas flow from combustion chamber to the crank case. In first ring designs, it was larger in diameter (app. 10%) than the piston. Thus, normal force by the pretension of the ring sealed off the gases. Further, the rings were modified to enable the combustion gas forces to act from the back of the rings along the ring groove. The more pressure on the back of the ring, the more sealing is provided [3].

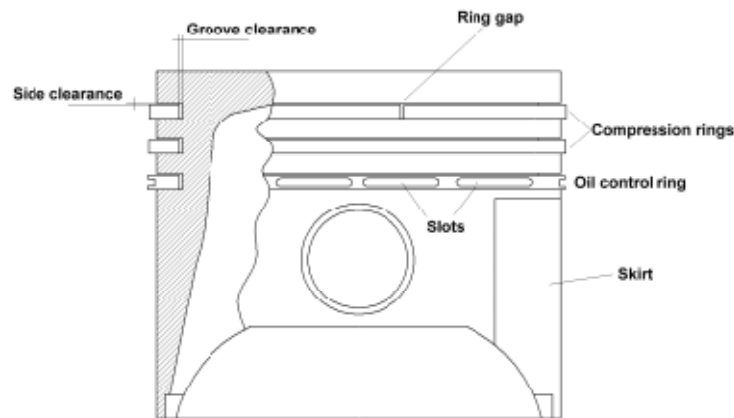


Figure 1.2: Piston and piston rings [3]

Sealing off the combustion gases is not the only use of the piston rings. Oil rings are also used to control the oil film thickness on the cylinder liner, which determines lubrication regimes. In the absence of sufficient oil on the surface, frictional forces and the wear on the liner will increase. However, the existence of a thick oil film results relatively higher oil consumption rates. The oil control rings distribute the sufficient rate of oil film on the surface and scrap off surplus oil to the crankcase [3].

The use of the rings can be listed as;

- Sealing of combustion pressure
- Distributing and controlling the oil
- Transferring heat
- Stabilizing the piston.

One of the phenomena is the blow by of combustion gases from combustion chamber to the crank case. In the absence of sufficient oil on liner-ring interface, the gases can pass through the front side of the oil, or the gases can pass from the back of the ring through the groove when the ring is not in contact with none of the either sides of the groove given in Figure 1.3.

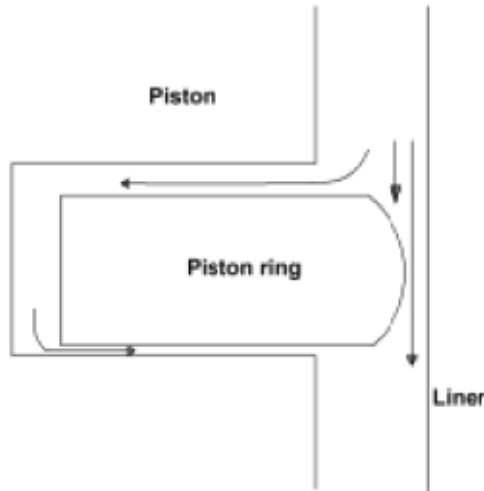


Figure 1.3: Combustion gas blow-by past the piston ring [3]

For the ring material, grey cast iron is commonly used for its beneficial properties as a dry lubrication effect of the graphite phase of the material in starved conditions. For the coating of the ring surface, chromium is usually used for its properties in corrosive and abrasive conditions.

The cylinders are usually made of cast iron containing phosphorus, manganese, chromium, molybdenum, vanadium and titanium as alloying elements, or steel or aluminum. The liner surface can be coated with a hard chromium layer to improve the wear resistance of the cylinder liners. Grey cast iron, when used for cylinder liners, is tribologically beneficial, as the graphite phase of the material gives a dry lubrication effect and furthermore acts as an oil reservoir that supplies oil at dry starts or similar conditions of oil starvation [3]. Surface porosity with chromium plating is also necessary for scuff resistance [4].

To limit hydrocarbon emissions and particles by reducing the oil consumption, the surface roughness of the cylinder liner should have R_a value between 0.25 and 0.4 and R_z value between 3 and 6. This can be achieved with optimally honed bore

surfaces. The surface finish of the cylinder liner has an influence on the scuffing resistance [3].

1.2 Piston Group Friction Mechanism

As a reciprocating piston mechanism, internal combustion engines have four main friction mechanisms affecting mechanical losses which are between piston and cylinder liner, piston rings and cylinder liner, and connecting pin, crankshaft, connecting rod friction. The losses due to piston, rings and rods are 40 to 55 percent of total mechanical losses [2].

Table 1.1: Power cylinder friction percentages [2]

	% Power Cylinder	% Total Energy	% Work Output
Piston/rings/Rods	100	1.6-8.3	4.1-20.9
Rods	18-33	0.29-2.7	0.74-6.8
Piston	25-47	0.4-3.9	1.02-9.8
Rings	28-45	0.45-3.7	1.15-9.4
Top	3.6-18.0	0.06-1.5	0.15-3.8
Second	2.8-9.9	0.05-0.8	0.11-2.1
Oil	14.0-33.8	0.23-2.8	0.58-7.0

Table 1.1 shows the distribution of the piston group friction losses for individual components. The piston and the rings cause 50% to 90% of the total power cylinder losses as shown in Figure 1.4 [2].

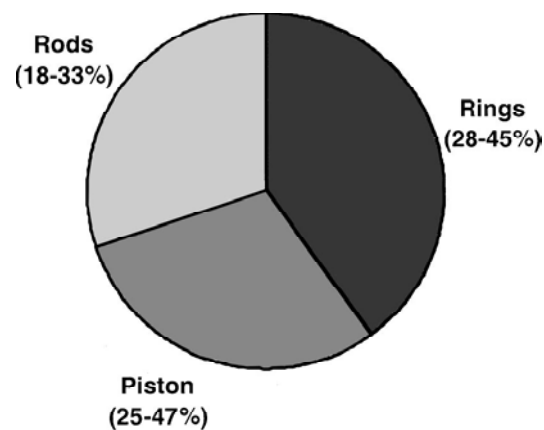


Figure 1.4: Distribution of piston/ring/rod friction

The main reasons of piston and the cylinder liner friction can be listed as;

- Clearance of piston and cylinder
- Surface roughness
- Piston skirt surface profile
- Surface temperatures and elastic and thermal deformations
- Piston material
- Lubrication regime and sufficiency of oil
- Piston secondary dynamics

The ring pack and the cylinder liner have a similar mechanism. The ring profile, surface roughness, lubrication quality, etc affect the friction coefficient. Since the ring tension directly affects the lubrication regime, it should be well optimized for sealing and friction. Also ring width, axial clearances take important place.

1.3 Friction Measurements of IC Engines

Measurement of friction in an engine assembly is a difficult procedure, since the friction values are relatively small compared to the indicated power inside the cylinder. Furthermore, it is hard to separate the contribution of individual components since there is more than one mechanism involved, In basic, removing components is a technique for specifying each components friction, but in practice, removing a component may not be so sufficient, due to the interactions of the components with each other. Additionally, difference tests can be considered to investigate the differences in individual designs [2].

In literature, there are several methods for engine assembly friction measurement. Friction Mean Effective Pressure (FMEP) and Brake Specific Fuel Consumption (BSFC) are two of these methods. In both cases, the differences of changes are tested and the little change in test conditions such as change in intake manifold temperature directly affects the test results. FMEP is the difference between Net Indicated Mean Effective Pressure (NIMEP) and the BMEP. In order to evaluate FMEP, cylinder pressures must be measured in order to find out NIMEP which is the produced power

of the engine. BMEP can be found by attaching a dynamometer to the crankshaft of the engine.

BSFC can be used when the cylinder pressure data is not available but it is really hard to get sufficient friction results from fuel consumption data because of the smallness of it in comparison with the output power [3].

Motoring tests are preferred rather than the other methods due to the easiness of these tests. A dynamometer is used to maintain the speed as constant and the torque of the dynamometer is recorded [5]. The difference in the torque is measured by removing individual components. In this method, the real combustion pressures and temperatures are not considered. Thus, pressurized motoring test are commonly used. The temperatures are not considered in pressurized motoring tests. As a consequence, firing/motoring tests take the place to simulate actual temperatures. In a Hot Shutdown test, the engine works under ideal conditions at first, then fueling to the injectors is stopped and the engine is motored, as the previous method [6]. Moreover, the engine is allowed to decelerate unlike the motoring tests in another method, so the deceleration curves are analyzed [7]. All of these methods need less instrumentation, however they need the entire engine for the tests and hence not flexible.

Besides the engine assembly test methods, more complex friction simulators have been introduced with much more instrumentation. Furuhami [8] modified an engine with a floating bore which is allowed moving in axial directions. This was an efficient method to see the effects of the operating conditions such as speed, viscosity, load, etc. on friction losses. However, in high speed operation, vibration problems were observed in the reciprocating liner tests [2].

Uras and Patterson developed Instantaneous IMEP method using strain gages on the connecting rod. They measured cylinder pressures and connecting rod forces and calculated piston assembly friction. The system was able to be motored with and without compression and firing [9].

In 1988, Ku and Patterson used Fixed Sleeve Method which was a modified version of floating bore method. In this method, a thin sleeve is placed between cylinder block and the floating liner, measuring the axial force on the sleeve and the gas force [10].

Besides these methods, a bench friction simulator is introduced by Akalin and Newaz, which has a reciprocating liner segment with a fixed ring holder carrying the strain gages. An external electric motor is used to give the reciprocating motion to the liner and the bending of the ring holder arm is measured for friction calculation. In their method, cylinder pressure is simulated as normal load on the ring and the temperature can be adjusted to the simulated test conditions as well as the speed and oil supply rate. Various types of rings and liners, also coatings and additives in oil can be tested with a wide range of operating conditions. [11-14]

1.4 Lubrication Regimes

Contacting of two sliding rough surfaces results three main lubrication regimes which are;

- Boundary Lubrication
- Mixed lubrication
- Hydrodynamic lubrication

In boundary lubrication regime, there isn't sufficient lubricant film on the contact region and the load on the surface is provided by asperities. This lubrication regime is occurred with high contact pressures and relatively low operating speeds which directly affect the life of the subjected gear, bearing, liner, ring, etc. The friction coefficient is maximum in such lubrication regimes so that the losses due to friction force increases. The asperity surfaces interact each other and heat is produced which may result a chemical reaction between the lubricant and the material. The interaction of the asperities in boundary lubrication is given in Figure 5.

In mixed lubrication regime, the lubricant film separates two sliding surfaces but still some contact between the asperities is observed. As a result, the load is carried both by the lubricant film and the asperity contact.

Figure 6 shows entire lubrication regimes including mixed lubrication regime. In this figure, Stribeck curve is also given. Stribeck curve is the relationship between dimensionless Sommerfeld number and the friction coefficient. Sommerfeld number is defined as;

$$S = \frac{U \times \mu}{P}$$

Where U is the speed in rev/s, μ is dynamic viscosity and P is the normal load on the contact surfaces.

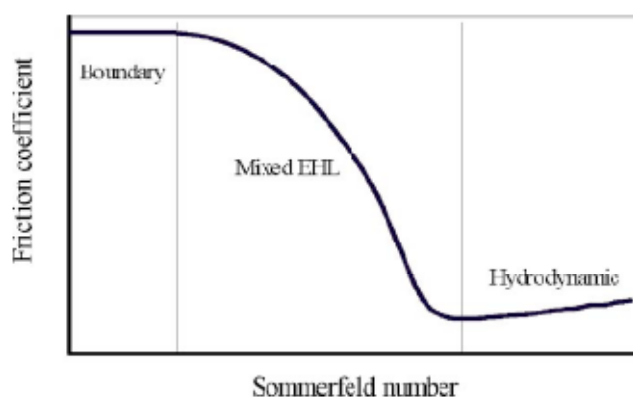


Figure 1.5: Stribeck curve and the lubrication regimes [15]

Increasing the speed in mixed lubrication results an increase in the lubricant film and the friction coefficient decreases due to lower load carried by asperity contact.

For higher speeds or higher oil viscosity, the film gets thicker between the surfaces and the asperities are separated from each other by the lubricant film. This lubrication regime is known as Hydrodynamic Lubrication. As it is seen in the figure, increasing the film thickness increases the friction coefficient due to viscous forces which start dominating the friction force.

In internal combustion engine applications, rings are generally in mixed lubrication regime during the stroke, but near at dead centers, due to low speeds, boundary lubrication may be observed. Hydrodynamic lubrication is only seen at cold starts. But the piston skirt has hydrodynamic lubrication properties due to lower loads at high temperatures.

1.5 Nanoparticle Additives

1.5.1 Background

Reduction in the power cylinder losses has been an important research topic for more than 30 years. Most of the factors effecting friction are limited by emissions, fuel consumption, performance and oil consumption. Lubrication quality takes great place in friction reduction. Recently, solid lubricants added into oil have been investigated

by several researchers, and their tribological behaviors showed a great revolution in surface technology and lubrication [16].

Lamellar sulphides, such as MoS_2 and WS_2 are used as solid lubricants and additives to oil, grease etc. result successful frictional behavior due to their lamellar structure. Although they have low friction coefficients, they are not available in so many applications, because the humidity and the O_2 in atmosphere greatly affect their chemical structure and they can only be used in space applications or in particular vacuum systems. Lamellar compounds of sulfides has tendency to stick to the mating surface due to reactive dangling bonds, they oxidize in a short time and lose their chemical stability [25].

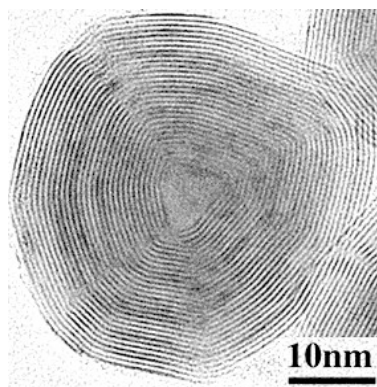


Figure 1.6: SEM image of IF nanoparticle [25]

Advances in fullerene technology, these lamellar sulphides were synthesized as nano-spheres, nested within each other. The spheres are like onions, which have about 15 layers and have no reactive edges causing oxidation in humid and O_2 in atmospheric conditions. They can be synthesized by MX_2 formulation, where M indicates Molybdenum or Tungsten; X indicates Sulfur or Selenium. These Inorganic Fullerene like nano particles are 40-200 nm in diameter; they are seamless, highly elastic and chemically stable [30]. They are used in lubrication, electron devices, catalysts, hydrogen storage bodies, super shock absorbers, photosensitive films and tips for scanning microscopy. As an additive to oil, the previous experiments showed more than 30 percent decrease in friction coefficient in various friction test systems [25-31].

1.5.2 Literature Survey on IF Nanoparticle Additives

1.5.2.1 Synthesis of Nanoparticles

A group of scientists suggested that layered metal dichalcogenides can form fullerene like structures. In literature, various synthetic methods are available; such as solid gas reactions, electron beam irradiation activation and arc discharge, thermal decomposition, hydrothermal synthesis, the sonochemical process, and template synthesis [23].

Tenne and co-workers produced MoS₂ fullerene particles by heating MoO₃ in an atmosphere of 95% N₂ and 5% H₂ at about 850 °C in order to break the oxide then by the reaction of oxide with a stream of H₂S mixed with forming gas. The product is MoS₂ nanotubes along with polyhedral particles [16]. Li et al obtained MoS₂ nanotubes and fullerene-like nanoparticles by the reaction of MoO₃ nanobelts and S in an argon atmosphere at 850 °C [19].

Synthesis of WS₂ is also made in a similar way. One of the methods in the literature is heating Tungsten in H₂S and H₂ in water vapor [20]. Another way is heating WO_{3x} particles in H₂S and forming gas mixture [21-22]. Taking Ti and Se powder as the starting materials, Chen et al synthesized TiSe₂ nanotubes/nanowires in an argon atmosphere at 650 °C for 8 hours [23]. By using the reaction between metal nanoparticles and sulfur powders in an atmosphere of argon and H₂S, Si et al synthesized nanocapsules with WS₂ (or MoS₂) shell material encapsulating CoS/CoO (or Mo) cores [24]. Nath et al produced WS₂ by thermal decomposition of ammonium thiotungstate at 1200-1300 °C with hydrogen flow. The reaction is given below [17]:



Yang et al prepared WO₃ nanoparticles from sodium-tungstate (Na₂WO₄) after a couple of chemical reactions. By preparing S and WO₃ nanoparticles, an amount of H₂ was introduced to the reactor and heated up to 900 °C, finally the IFs were available as the product [16].

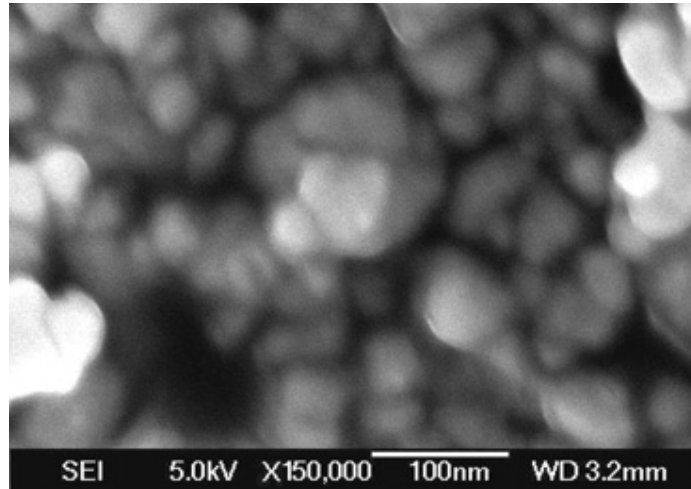


Figure 1.7: High-magnification FESEM image of as-prepared WO_3 nanoparticles[16]

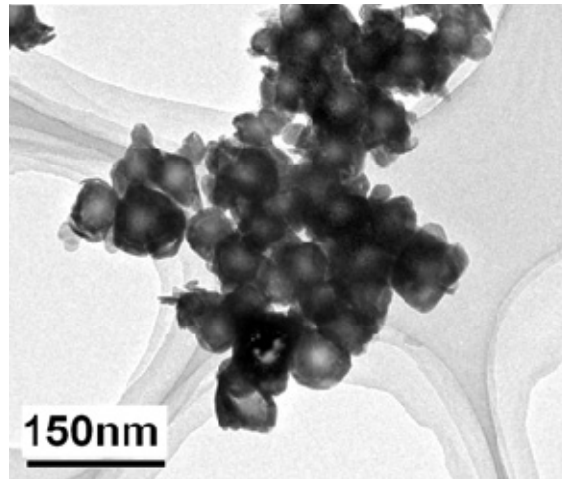


Figure 1.8: Typical HRTEM of synthesized IF- WS_2 nanoparticles annealed at 620 °C for 30 min. [16]

After a decade of inventory, different types of IF materials can be synthesized. Most kinds of MX_2 type transition dichalcogenides (Table 1.2), transition metal oxides (TiO_2 , V_2O_5 , CS_2O , Ti_2O), halides (NiCl_2 , CdI_2), elemental nanotubes (Bi,Te) of IFs are now available [16].

Table 1.2: Transition dichalcogenides type IFs

M	X₂
Tungsten	Sulfur, Selenium
Molybdenum	
Tin	
Thallium	
Rhenium	
Niobium	
Tantalum	
Hafnium	
Zirconium	

1.5.2.2 Experimental Studies on IF Nanoparticles

Additives in oil, grease, etc., are used widespread for improved lubrication characteristics. MoS₂ and WS₂ platelets are the most common additives, due to the superior frictional behaviors of these molecules [27]. In contradiction, these platelets have reactive edges causing tendency to stick to the mating metal pieces, which cause oxidation [25]. Besides, by the improvement of fullerene technology on carbon systems, first metal dichalcogenides MX₂ (M=W,S; X=S, Se) were synthesized [25,27,28]. In past 10 years, these nano scale additives are investigated by many researchers.

Cizaire et al. [25] investigated the tribological behavior of IF-nanoparticles as an additive in oil and as coating on the surface in ultra high vacuum conditions. After the tests, the HRTEM studies showed that, IF-nanoparticles maintained their chemical properties, but they were physically flattened or broken in individual sheets. Rapoport et al [26] analyzed the mechanisms of friction of IF in oil and in the powdered samples by a ring-block tester at various speeds and loads. They obtained

low friction values in relatively low loads. In low speed and load operations, IF remained undamaged so the rolling and suspending of IF is observed, but increasing the load, and also the speed, the particles were broken and the friction coefficient was increased.

Rapoport et.al [27] in 2003 tested IF nanoparticles under mixed lubrication regime on pin-on-disk tester, found out that instead of rolling of IF, the transfer of the broken sheets are more dominant in friction reduction. They observed an film covered the surfaces of the pin, so the wear rate at extreme loads is lowered.

Rapoport et al. [28] showed that the impregnation of IF into porous matrices has better results than impregnation of 2H solid lubricants. Release of nano spheres from the pores during the operation decreases the ploughing effect and provides fresh solid lubricants to the contact area [28, 29]. They analyzed the wear debris which is seen to be surrounded by spherical IF, so the friction coefficient and the wear rate is improved [28].

Greenberg et al. [30] used three different test methods, which are flat on flat, rib on flat, and ball on flat tests. On flat on flat tests, due to lower loads and shorter time of sliding, adhered IF particles on the surface is limited and so the effect of IF negligible. On the other hand, increasing load in rib on flat and ball on flat tests, friction coefficient seemed to decrease 40 percent in mixed lubrication regime. Rapoport et al. used higher loads and obtained no effect of IF on friction reduction when the film thickness is lower than IF size [31].

1.6 Surface Characterization

1.6.1 Methods

There are surface characterization methods in literature for imaging, determining elemental composition, microstructures, crystallography, etc. given in Table 1.3.

Table 1.3: Surface Analysis Methods [35]

Desired Information	Techniques
Imaging, profilometry and/or quantitative measurements of film thickness	Atomic force microscopy (AFM)
	Scanning tunneling microscopy (STM)
	Transmission electron microscopy (TEM)
	Scanning electron microscopy (SEM)
	Scanning transmission electron microscopy (STEM)
	Variable angle spectroscopic ellipsometry (VASE)
	Light microscopy (RBF, FM and CLSM)
Elemental composition and chemical State Measurements	Auger electron spectroscopy (AES)
	Energy-dispersive X-ray spectroscopy (EDS)
	X-ray photoelectron spectroscopy (XPS)
	High-resolution electron energy-loss spectroscopy (EELS)
	Fourier transform infrared spectroscopy (FTIR)
	Fourier transform Raman spectroscopy (FT Raman)
	UV-Visible spectroscopy (UV/Vis)
Microstructure, crystallography, and defects measurement	High-resolution transmission electron microscopy (HRTEM)
	Low-energy electron diffraction (LEED)
	X-ray diffraction (XRD)
Real-time measurements of Surface Interaction	Surface Plasmon Resonance

1.6.2 Raman Scattering Method

Raman scattering is the inelastic scattering of a photon discovered by C.V. Raman in 1922. This method is used for identification of unknown compounds, chemical and bonding states, phase transitions, imaging and mapping of the material.

Raman scattering is based on measurement of the scattered photons from the surface of a sample which has been subjected to a strong monochromatic laser source. According to Rayleigh, when light is scattered from a molecule or an atom, the photons are elastically scattered which means maintaining their energy and wavelength. This phenomena is called Rayleigh Scattering. However, a fraction of the scattered light, which is really low, is scattered by an excitation, having different

frequency levels from the sent light. This is called as Raman Scattering. The intensity of the scattered light in Rayleigh scattering is $10^4 - 10^5$ times more than it is in Raman Scattering. However, Rayleigh scattering gives only one peak but it doesn't give any idea about vibration transitions. In Raman Scattering, the energy difference between the sent light and the scattered light is in the same range with the energy level difference of the vibrating molecules or atoms which are exposed to the light. So, information about vibration energy levels of molecules can be found by analyzing the Raman Scattering, which is called Raman Spectroscopy. In this method, the differences of wavelengths of sent and scattered light is measured. These differences are called as Raman Shifts [34].

In Figure 10, the relation of Raman scattering with the vibration energy levels is shown. When an unabsorbed photon with $h\nu_0$ energy level collides with a molecule, a few of them transfer the energy to the molecule after scattering or a few of them are transferred the energy from the molecule. As a result of this energy transfer, the molecules are in different energy levels after the collision.

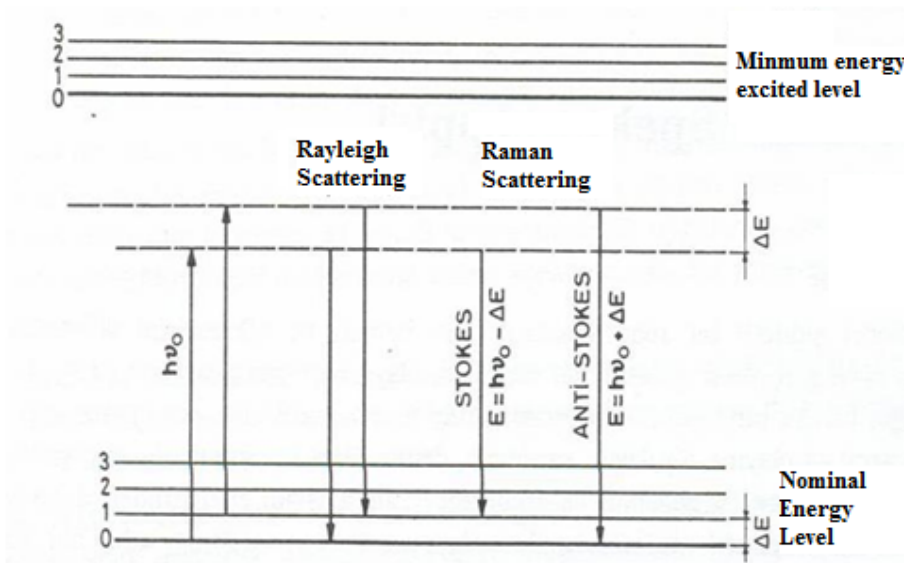


Figure 1.9: Energy levels in scattering technique [34]

Raman is an efficient method for analyzing unknown materials. One of its great advantages is reacting for non metal molecules or atoms, so that analyzing the tribofilm on the surface of the samples is possible. After scanning an interval of wave numbers on a sample, some of the wave numbers give peak values which differs every molecule or material from each other. The identification of the material is made by comparing the material library with that analysis.

In Figure 11 and Figure 12, Raman spectrum of different materials is shown.

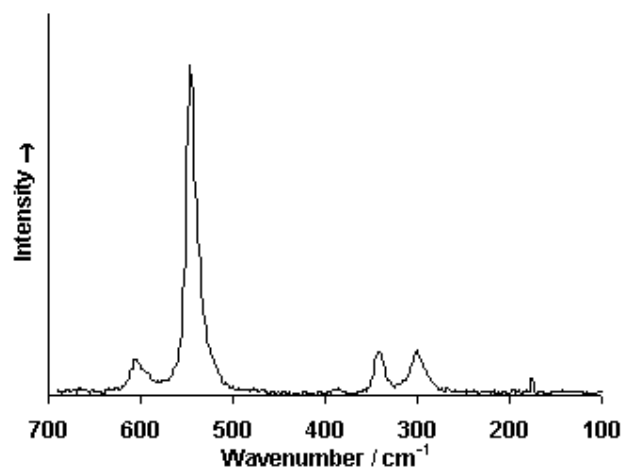


Figure 1.10: Raman Spectra of Cr_2O_3 [35]

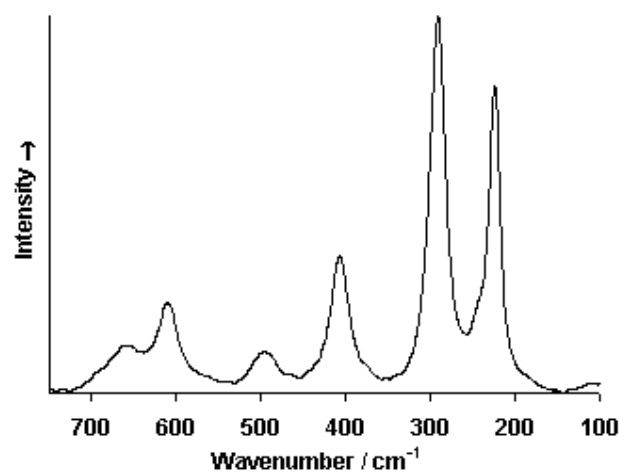


Figure 1.11: Raman Spectra of Synthetic Fe_2O_3 [35]

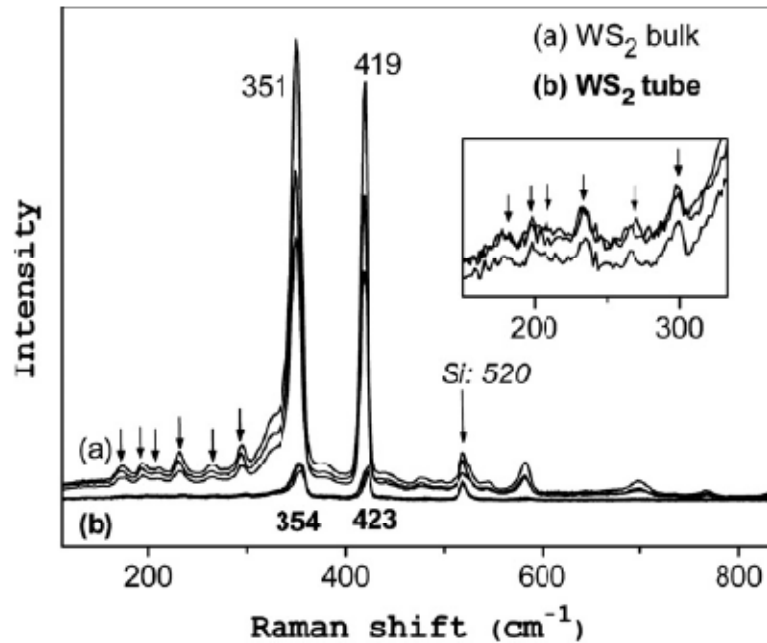


Figure 1.12: Raman spectra of WS₂ bulk and nano tubes [33]

1.7 Objectives of the Study

In this study, inorganic fullerene like nanoparticles added into mineral oil will be tested with a friction simulator with realistic engine temperature, speed and loading conditions. Friction data will be collected for different concentrations of IF nanoparticles and the effects of the concentration will be investigated. IF nanoparticles have been reported in many researches giving great results on friction reduction, and it is needed to be investigated in simulated engine conditions to discover the performance as an additive in engine oil.

The friction mechanism will be analyzed with different speed and loading conditions. The major reasons of the friction reduction will be discussed.

A detailed surface analysis with Raman Spectroscopy will be applied for the tested surface, which will give idea about the potential film formed by the use of these nanoparticles.

Also the use of the nanoparticles in internal combustion engine applications will be discussed for performance and fuel economy.

2 EXPERIMENTAL PROCEDURE

The test system designed and built by Akalin and Newaz, in 1998 is improved and adapted to test IF nanoparticle formulated oil's tribological performance. It is a reciprocating test system with high stroke capability, which controls most of the operating conditions such as speed, load, temperature, oil supply rate, etc. Real engine liner and ring portions can be tested; also different type of oils can be simulated with realistic speeds. Once the test runs; friction force, load and the operating temperature are recorded with every crank angle by a multiple channel data acquisition card simultaneously. One of the most important features of the system is that the system allows running in hydrodynamic lubrication regime region due to high speeds and higher strokes unlike other simulated test rigs.

2.1 Measurement System and Instrumentation

2.1.1 Electric Motor & Crank-Connecting Rod Mechanism

The excitation of the test system is achieved by a 1.5 HP permanent magnet servo motor with a speed drive unit. It has its own encoder inside for on/off switching, as well as an output channel for encoder signals and an input channel for speed control with analog voltage. During the tests, the speed is controlled by analog voltage via the input channels in a range of 0-5 V contributing to 0-2000 rpm. The crank is directly connected to the motor shaft without any speed converters.

When the crank is activated, the rod pushes and pulls the liner holder, giving the reciprocating motion to the liner sample. In the system, piston is designed to be stationary while the liner is sliding. The counter weights are oppositely connected to the crankshaft to balance the weights of the mechanisms. Therefore the first and second harmonic inertia forces are balanced.

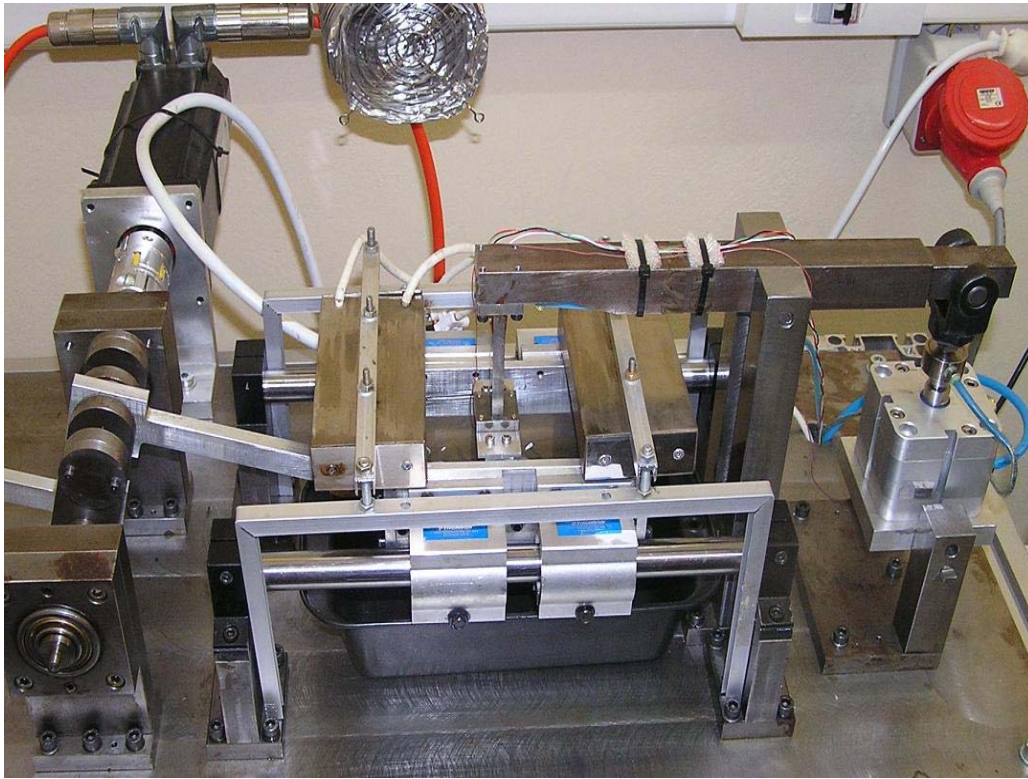


Figure 2.1: Reciprocating bench test system

Two rolling bearings are used for each sides of the liner holder to provide low maintenance for the bearings. The connections of these rolling bearings also allow adjusting the height of the liner holder to satisfy the uniform clearance between the ring and the sample during the entire stroke.

2.1.2 The Ring Holder

The ring holder meets the ring to the cylinder liner, limiting the axial and lateral motion of the ring. Furthermore, normal load is transmitted to the ring by the ring holder. Aluminum alloy is used for the ring holder to decrease the weight/strength ratio. There is a groove in the middle of the holder for the ring seen in Figure 2.3. This groove's tolerance is important for the motion characteristics of the ring inside the holder. Higher groove dimensions will result the tilt motion of the ring, while low dimensions will restrict the ring inside the holder so the lubrication characteristics will be changed. The ring segment provided by Federal Mogul has a 2.385 mm width. The ring groove is opened 2.42 mm width as well. Pin couples in the edges are restricting the ring for the lateral motion while the ones in the middle transmit the

load. The plate upside is placed for the assembly connection of the holder to the holder arm.

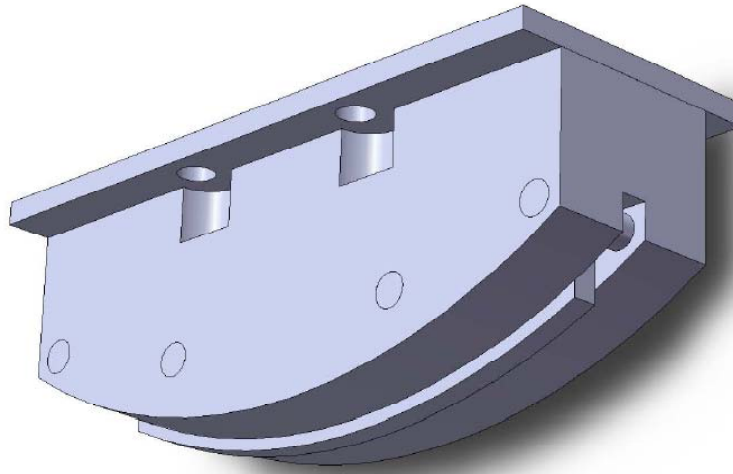


Figure 2.2: 3D model of the ring holder and the ring

2.1.3 The Air Cylinder

In internal combustion engines, the pressure inside the cylinder applies force to rings from the back pushing them to liner. In the test system, the effect of the load is simulated by a compressor - air cylinder approach. The compressor pumps air to the air cylinder which converts the pressure to applied force seen in Figure 2.4. Air pressure is adjusted and regulated by the air cylinder. A load sensor is placed between the loading arm and the air cylinder for dynamic load measurement.

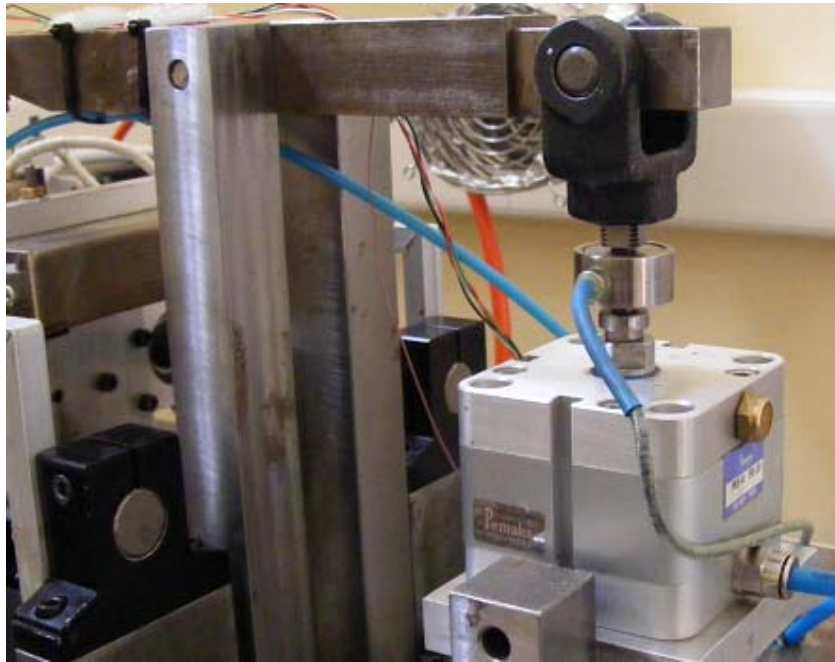


Figure 2.3: The air cylinder

2.1.4 Friction Force and Normal Load Measurement

Instantaneous friction force is measured by a strain gage circuit in full-bridge configuration shown in Figure 2.1. As the liner reciprocates, the friction force between the liner and the ring bends the arm. The strain gage circuit measures the bending and produces a voltage in the terminals of the circuit. This voltage is acquired by a data acquisition card for every crank angle.

Normal load is measured by an Omega load sensor. Maximum load measured by the sensor is 450 N [11].

The voltage levels produced by the sensors are exactly too small. In order to acquire the signals significantly, VISHAY A2 signal conditioner is used.



Figure 2.4: Signal conditioner

A programmable filter is used to block the noise components of the signals. A Bessel filter with 150 Hz cut-off frequency is used. Signal cables are then connected to NI™ SCB-68 shielded connector block which is attached to data acquisition card.

National Instruments DAQ-Card 6062E is used in data acquisition system. The DAQ card has 8 analog input voltage channels two of them of which is used for normal load and friction force measurements. Moreover, there are two analog voltage output channels, one of which is used for sending voltage signals to the drive unit for speed control of the motor.

One of the most important channels is the external source (trigger) channel. The signal can be collected by an interior clock in continuous time domain or by available external pulses. Signal is acquired for every crank angle by triggering from the external clock in this system. The driver of the servo motor produces signals for every crank angle, so the DAQ card acquires friction and load signals when the crank angle signal is received.

NI LabView software is used for data acquisition. A user interface is designed to collect, post process data, and also to adjust the speed of operation. The user interface is given in Appendix 1.

For observation, the data is collected continuously and monitored by a scope seen at the top in Appendix 1. By clicking 'save' button, the continuous acquisition stops but 10 cycles of data acquisition for recording starts. There are MATLAB© scripts in the block diagram of the program, which organize the collected data. For the experiment, the data is collected for 720 CA meaning a whole four stroke engine cycle. In order

to minimize the noise, the acquisition process is repeated for 10 times, and then the averages of 10 cycles for every crank angle are executed. After acquiring 7200 CAs, the averages are taken and monitored by additional scopes. The written codes can also edit the saved data; compare the experiments to see the differences of individual tests.

2.2 Experimental Setup

Cylinder liner samples cut from cylinder liners are provided by Federal Mogul, Turkey. The surface characterization of individual liner segments is investigated before the tests. The surface roughness parameters, such as average roughness R_a , root mean squared roughness R_q , maximum height of the profile R_t , skewness R_{sk} , etc. are measured.

The rings are also provided by Federal Mogul Turkey, and they are made of chromium plated steel. The rings are cut to 50.8 mm length to fit the liner sample (2 inches). Hexane solvent is used to clean the surfaces of ring and liner samples.

The plain oil DX-30 used in experiments is provided from ApNano Materials Inc. as well as the IF-formulated oil. The reference oil is Pazmular DX-30 engine oil, with 12 cSt kinematic viscosity at 100 C.

IF nanoparticles formulated oil was received 10 % in concentration. Three different solutions in different concentrations were prepared, which are 1 %, 5 %, 10%. In order to see the effects of the concentration, different concentrations of IF formulated oil are tested through the test matrix.

The experiment of IF nano particle added engine oil consists of six main parts.

- Run-in test with reference oil
- Test matrix operation for 20 and 70 C
- Run-in with IF-added oil
- Test matrix operation for 20 and 70 C
- Returning to base oil run-in
- Re-testing the test matrix

Running in test is the first step of the experiment. In order to get rid of surface roughness variations during the experiment, the system should be run in a definite

speed and load for a specified time interval. This time interval is assigned through the loading and speeding conditions of the samples, oil type and the material properties. During the test, the friction data is collected by every 10 minutes and the running in process is continued until the change in the average friction coefficient is stabilized by 1%. Due to the high roughness values of untested samples, friction coefficient values are predicted to be slightly higher for the first results.

After the running in period, reference oil tests with specified test matrix take place. The speed and load are adjusted for each test to take the reference oil matrix results. In this step, load is adjusted to 80 N at first, and then the speed is changed from 300 to 700 rpm. At each point, the results are acquired at the same temperature.

After taking the reference oil matrix data, the surfaces of the ring and liner segments are washed with hexane solvent to clean surfaces from previous test's oil and third body wear particles mixture. The syringe and the oil feeding pipe is changed for new type of oil. The IF nano particle test includes 3 concentrations mentioned before. At first, the effect of low concentrated oil is investigated, so the syringe is filled with 1% IF formulated oil. For the following experiments, the other concentrations are tested, respectively.

Raman scattering method used for surface analysis. The micro Raman system (Jobin-Yvonne HR 800) uses He-Ne laser beam with a wave length of 632.81 nm and focused to a spot size of 1-2 μm . The Raman analyzer program did not include the libraries of WS_2 compounds, so the past studies with WS_2 mentioned in the first section is used to compare the results for remarking the existence of WS_2 film.

Before the Raman analysis, the surfaces are cleaned with hexane solvent in order to get rid of oil and dust from the surface.

Firstly, the camera of the system is opened for focusing to the subjected area of the sample. After the alignments, scanning of the wave numbers from zero to 1500 cm^{-1} is executed.

2.3 The Test Matrix

The reference and IF nanoparticle formulated oil tests are made following a test matrix given in Table 2.1. In the table, four main speed levels are seen. The lowest speed test is at 300 rpm due to noise and scuff problems. According to Stribeck

diagram, as the speed decreases, the friction coefficient increases in mixed lubrication regime. The speeds lower than 300 rpm at relatively high loads causes stick-slip motions of the ring which corrupts the surface. Besides, higher speeds cause extra vibrations on the test system. So the speed range is specified between 300 – 900 rpm.

Normal load is chosen between 80N – 320 N. Under 80 N, noise is more dominant than the signal, while values higher than 320 N are not available for this instrumentation.

Table 2.1: 70 °C Oil Temperature Test Matrix

Speed \Load	80 N	160 N	240 N	320 N
300 rpm	x	x	x	x
500 rpm	x	x	x	x
700 rpm	x	x	x	x
900 rpm		x		

Table 2.2: 20 °C Oil Temperature Test Matrix

Speed \Load	80 N	160 N	240 N	320 N
300 rpm	x	x	x	x
500 rpm	x	x	x	x
700 rpm	x	x	x	x
900 rpm		x		

3 EXPERIMENTAL RESULTS & DISCUSSION

3.1 Reference Oil Results

In Figure 3.1, the friction coefficient graph for standard test condition (160 N, 500 rpm, °C) is shown. This type of frictional characteristic is called u-n type graphs, due to its shape. As the speed turns negative after 180 degrees, the friction coefficient takes negative values as well. At dead centers, friction coefficient is zero due to zero sliding velocity. The friction coefficient values start from top dead center, which means 180 CA degrees is the bottom dead center, and turning back to top dead center at 360 CA degrees and so on.

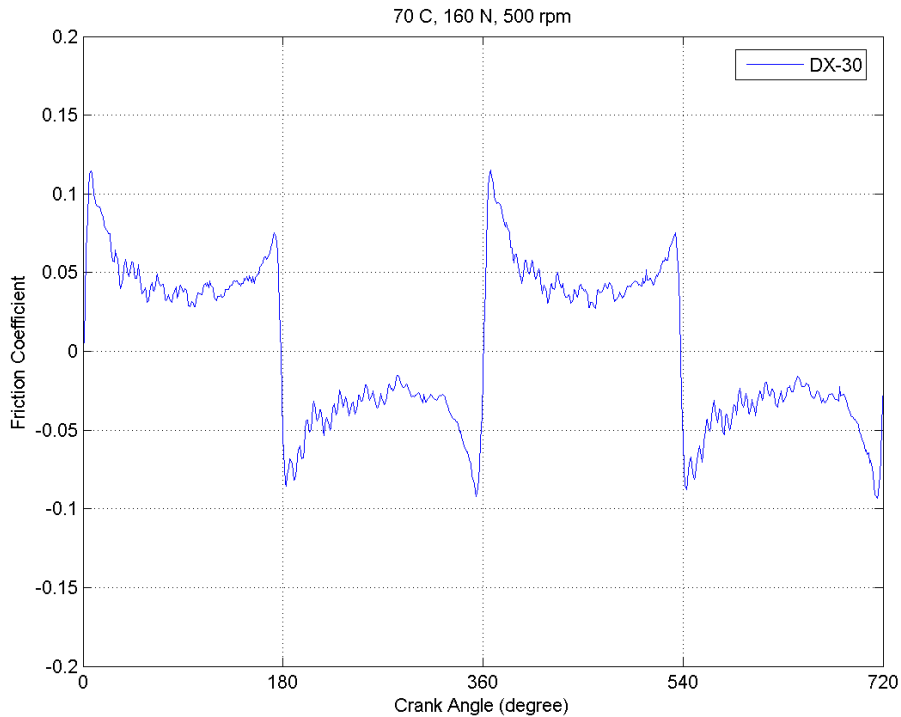


Figure 3.1: Base oil Test Results

Near at the dead centers, sliding velocity decreases sufficiently and the film thickness between the ring and the cylinder liner decreases either. As the film thickness decreases, the load carried by the oil film drops and the friction coefficient increases. At the middle of the stroke, sliding velocity is the highest so the film thickness

increases. Therefore, a drop in friction coefficient is observed in mixed lubrication regime.

Also the speeds at the dead centers are not equal. As a result, the friction coefficient values at the top and the bottom dead centers may differ.

The oscillations through the stroke may be because of the electrical noise or the oscillation of the normal load and also may be the result of test system components' self vibrations.

In Figure 3.2, the run-in process of new cylinder liner and ring segments are shown. The system is run in at 160 N normal load at 500 rpm, which is the nominal test conditions. The data is acquired by 10 minutes time friction coefficient for every cycle is averaged. After 90 minutes of run, the average friction coefficient seems to stabilize, nearly at 0.055 friction coefficient values. This process is needed to eliminate the effect of surface roughness changes during the running in period.

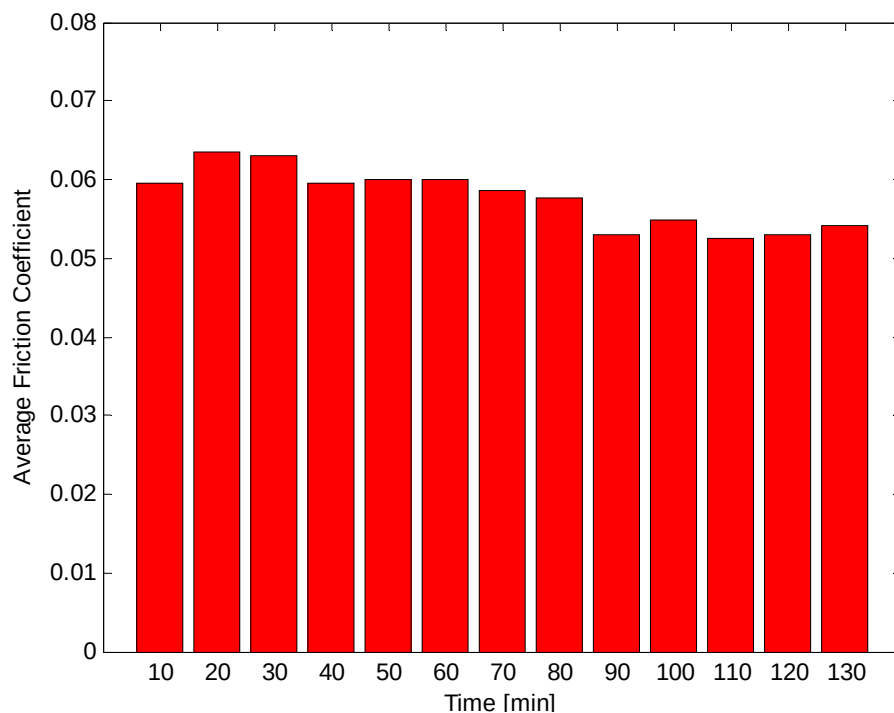


Figure 3.2: Base Oil Run-in Test

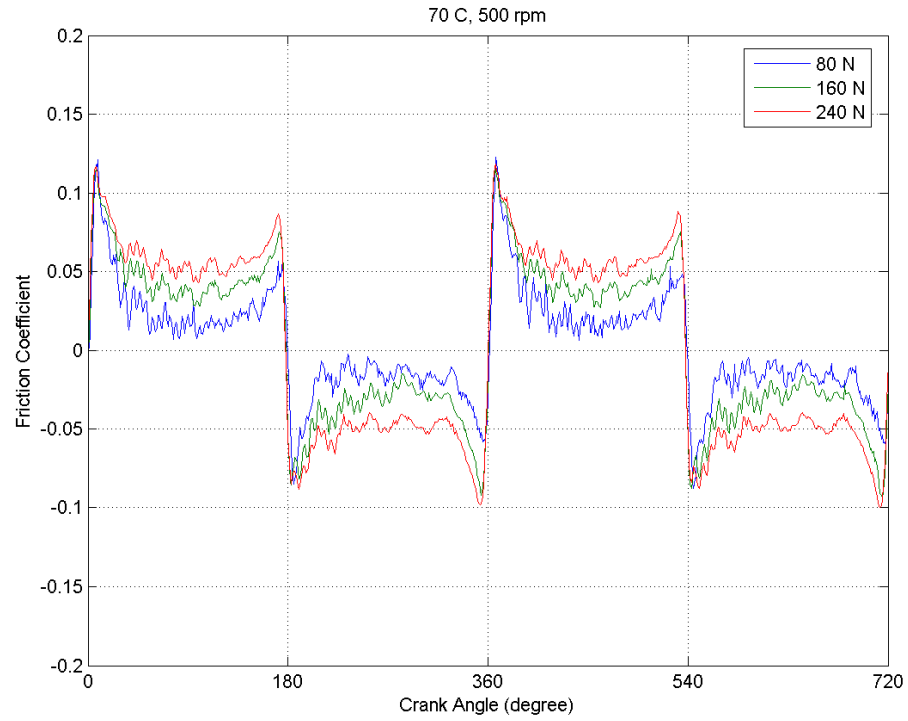


Figure 3.3: Effect of load on friction coefficient

In Figure 3.3, the effect of load on friction coefficient between the liner and the ring segments is shown. Both three tests are made in 70 °C at a constant speed of 500 rpm.

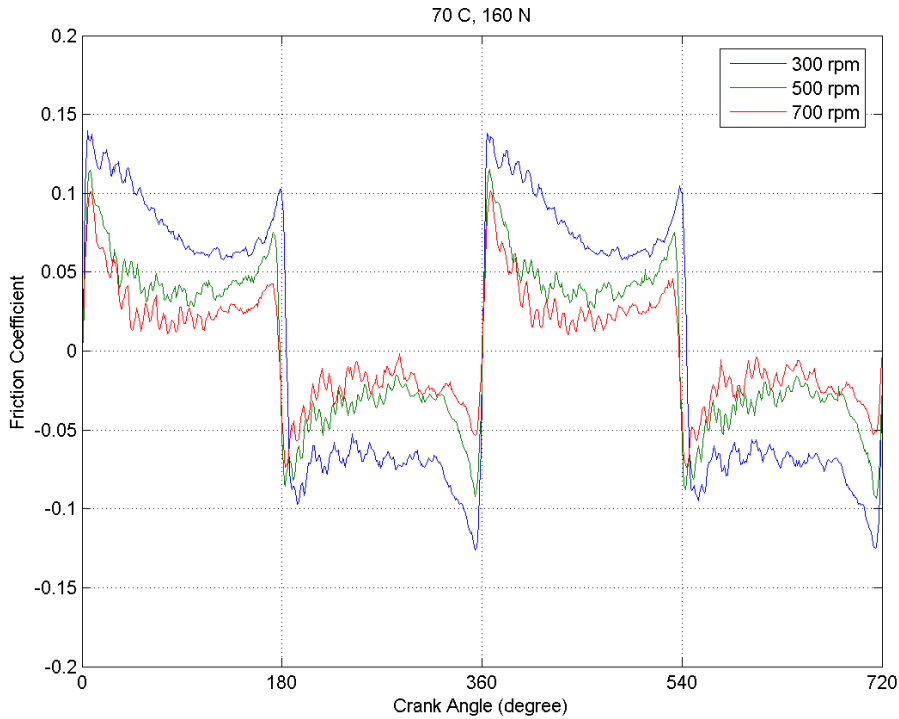


Figure 3.4: Effect of speed on friction coefficient

The effect of the speed is given in Figure 3.4. As it is seen the lower speeds gives higher friction coefficient values. Increasing the speed causes a rise in oil film thickness which results lower friction coefficients due to higher Sommerfeld Numbers in mixed lubrication regime mentioned in chapter one.

In internal combustion engine applications, the temperature of cylinder liners is limited by cooling process and the cooling water temperature can be taken as cylinder liner temperature in steady state, practically. As the temperature rises, the viscosity of the oil drops sufficiently, nearly 5-6 times; and by the effect of normal load, lubrication properties between the liner and the rings seems as mixed lubrication regime for entire stroke. However, the lubrication regime between piston skirt and the cylinder liner is usually hydrodynamic. Cold (20 °C) tests are made to see the effects of IF nano-particles in hydrodynamic lubrication regime. At 20 °C, the viscosity rises and hence the film thickness. In Figure 3.5, 20 °C test with 160 N normal load and 500 rpm speed is shown.

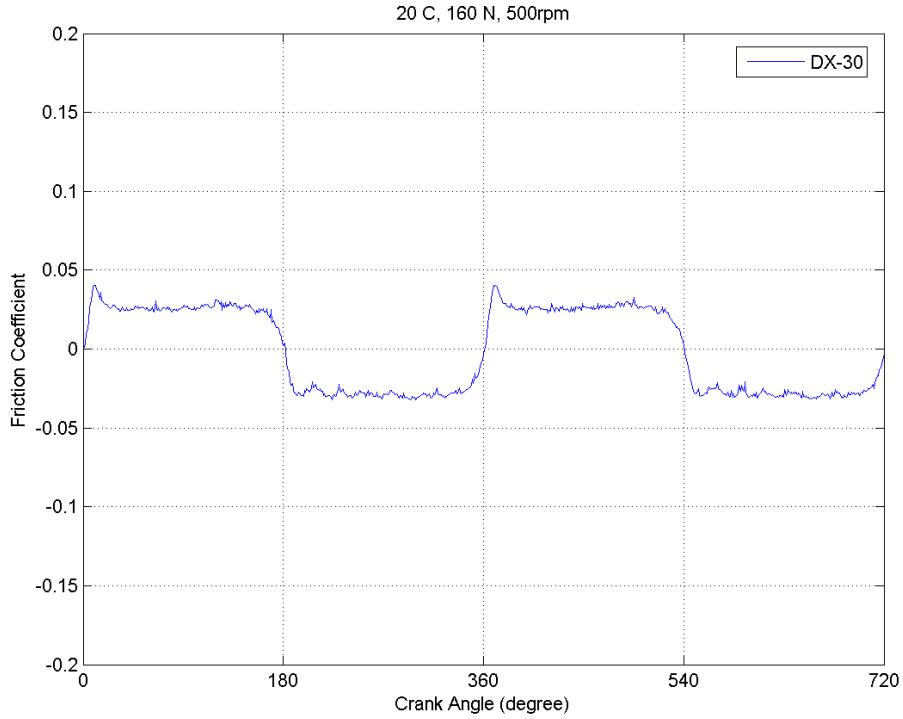


Figure 3.5: Hydrodynamic lubrication regime in cold operation

Unlike the results of 70 °C, 20 °C tests showed smoother friction coefficient values. Increase in the viscosity of oil by 5 times, it becomes thicker. The change in friction coefficient defined by Sommerfeld is given in Eq. 1. As the viscosity is multiplied in cold application, Sommerfeld Number increases and the lubrication regime becomes hydrodynamic according to the Stribeck curve given in Figure 1.6.

3.2 1% IF-Oil Mixture Results

After the run-in operation, the reference test data were collected according to test matrix given in Table 2.1 and 2.2 in the previous section. The next step is the testing of IF nano particle added into DX-30 reference oil in different concentrations. The surfaces of the ring and the liner segments are cleaned with hexane solvent before the IF tests in order to avoid the effects of wear particles and the used oil on the results.

1 % IF concentrated oil running in results are given in Figure 3.6. The run-in tests with 1 % IF is made under 160 N normal load with a speed of 500 rpm at 70 °C. The average friction coefficients are collected for 240 minutes by 10 minutes time intervals. First part of the plot is the 140 min run-in operation of the sample with reference base oil. As it is seen in the figure, the average friction coefficient does not

seem to fall after 240 minutes of operation with the use of 1 % IF concentrated oil. However, a sudden rise in friction coefficient is observed for the first twenty minutes. This rise is followed by a drop about half an hour of run.

This sudden rise is observed in the all IF tests. This is because of the behavior of IF nano particles as third body particles in contact surface in the absence of tribofilm on the surface.

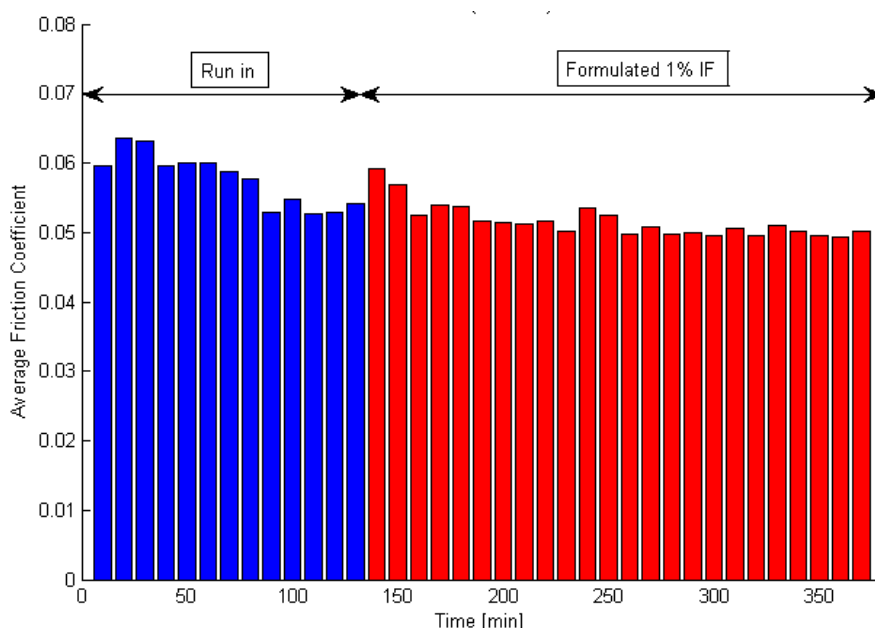


Figure 3.6: 1 % IF Experiment run-in results under 160 N with 500 rpm speed

In Figure 3.7 and Figure 3.8 below, show 70 °C experiments in 500 rpm. The blue lines are indicating the experiment with reference oil, while the green ones are 1 % IF added oil test after 140 minutes run-in, and the red line is again the reference oil after cleaning of the surfaces with hexane solvent. In these figures, it is seen that IF nanoparticles added into oil in 1 % concentration did not reduce the friction coefficient, even after 240 minutes of run. It is thought that the potential film formed on the surface is not sufficient to reduce the friction coefficient, due to low concentration of IF nanoparticles in oil.

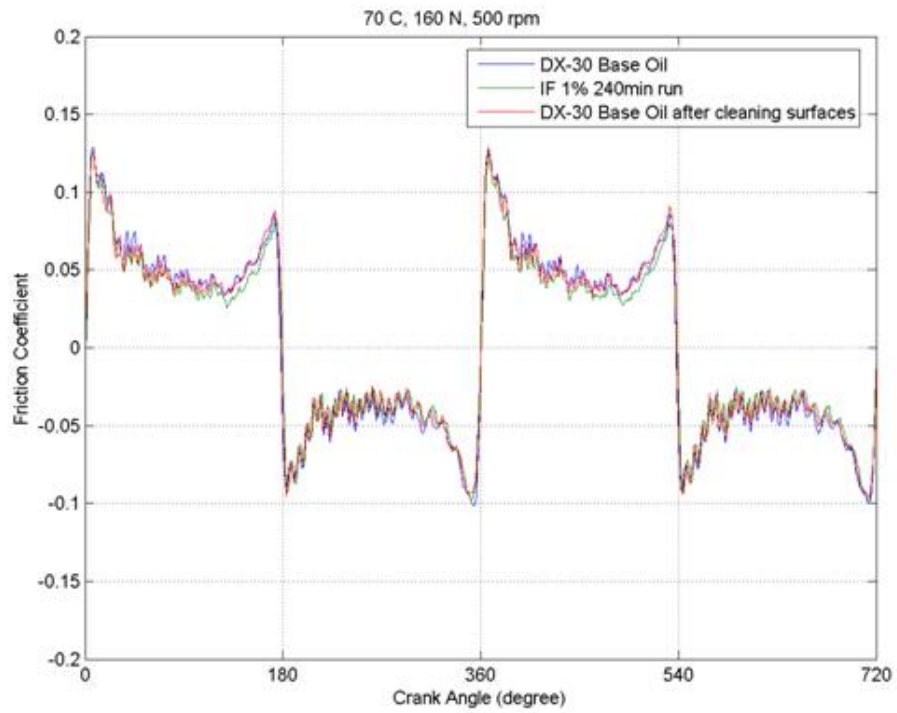


Figure 3.7: 1 % IF oil comparison results at 70 °C, 160 N and 500 rpm

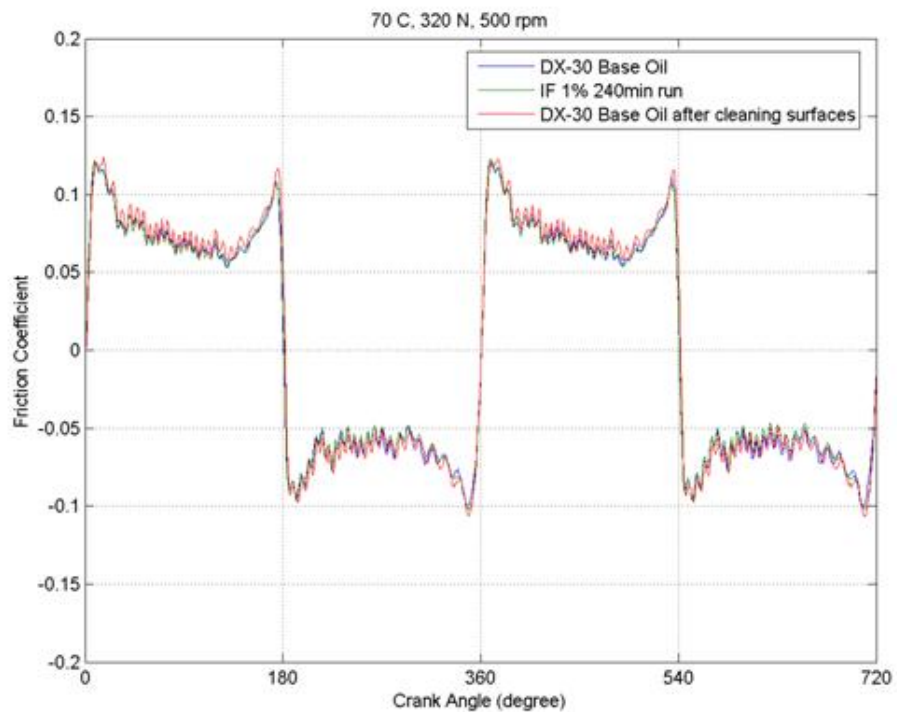


Figure 3.8 : 1 % IF oil comparison results at 70 °C, 320 N and 500 rpm

The formation rate of tribofilm on the surface of the liner is proportional to IF nano particle concentration in oil. As the ring slides on the liner, a matter of WS_2 spheres break into layers and form a thin film on the surface; however the scratching of the formed film is occurring continuously. This concentration is not enough for a reduction in friction coefficient for ICE applications.

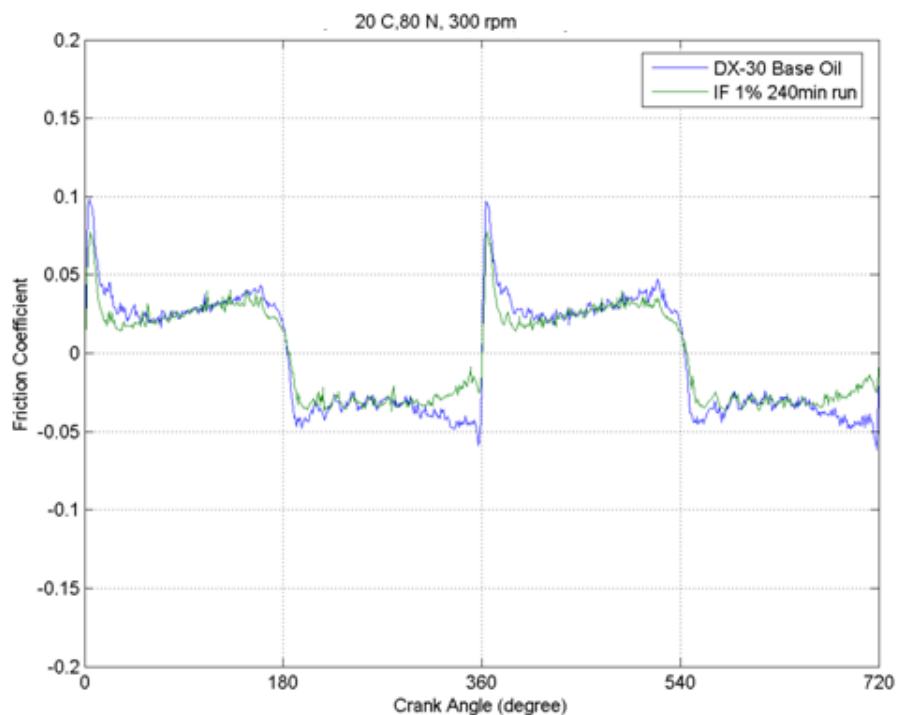


Figure 3.9: 1 % IF oil comparison results at 20 °C, 80 N and 300 rpm

Figure 3.9 and Figure 3.10 shows 20 °C experiments are shown. The blue lines show the reference oil test while the green ones are tests with 1% IF oil results after 240 minutes of IF oil operation.

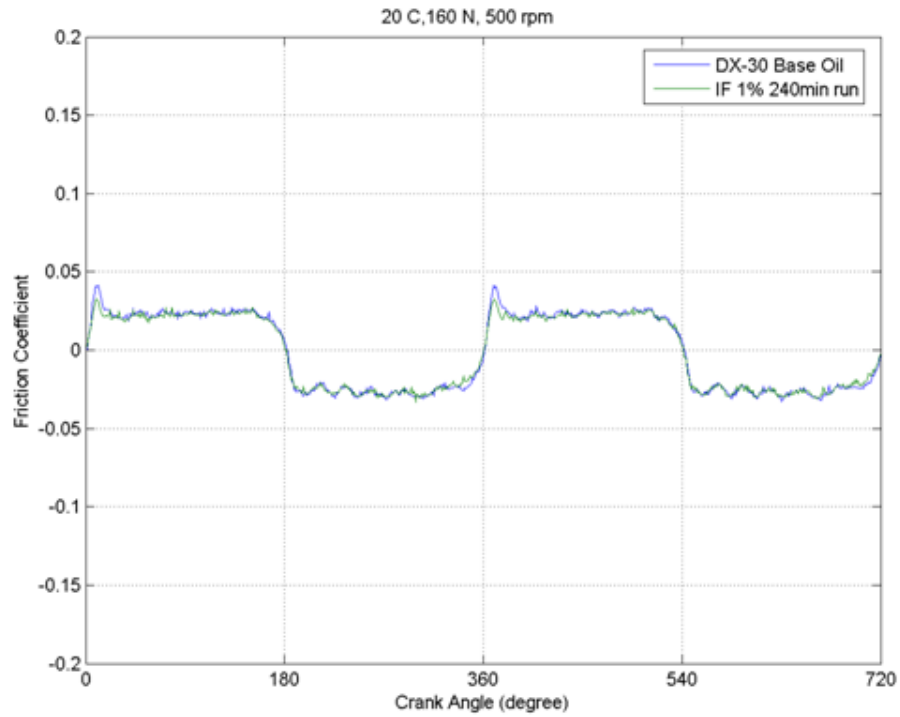


Figure 3.10: 1 % IF oil comparison results at 20 °C, 160 N and 500 rpm

3.3 5 % IF-Oil Mixture Results

The running in test results of 5 % formulated oil is shown in Figure 3.11. After the running in period, the reduction in friction coefficient was not more than 5%. This concentration showed nearly no effect on average friction coefficients.

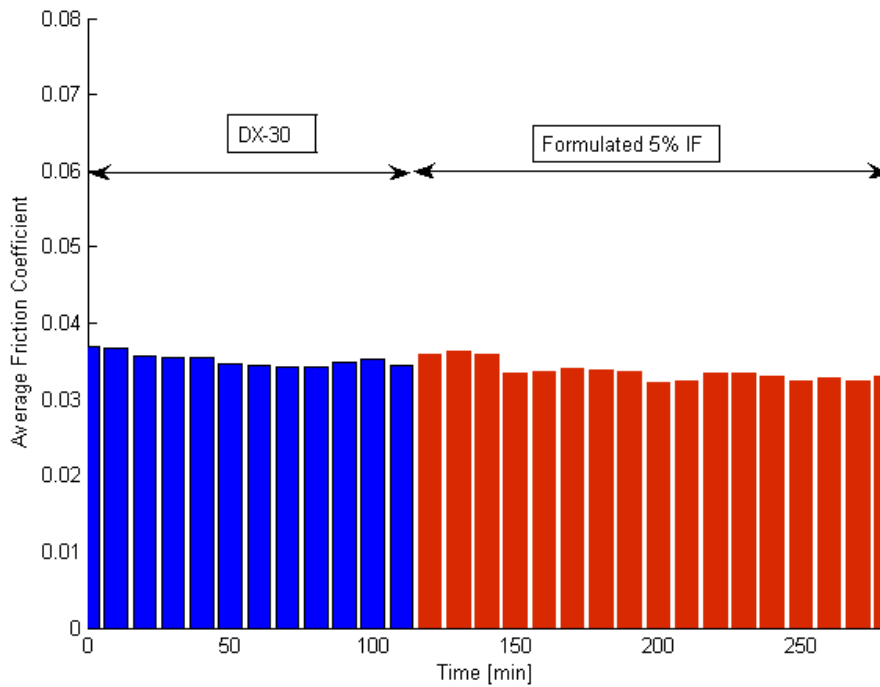


Figure 3.11: 5 % IF Experiment running in results at 160 N with 500 rpm speed

Figure 3.12 shows the results of 5 % IF formulated oil at 160 N and 500 rpm. There is not a great effect of IF on friction coefficient.

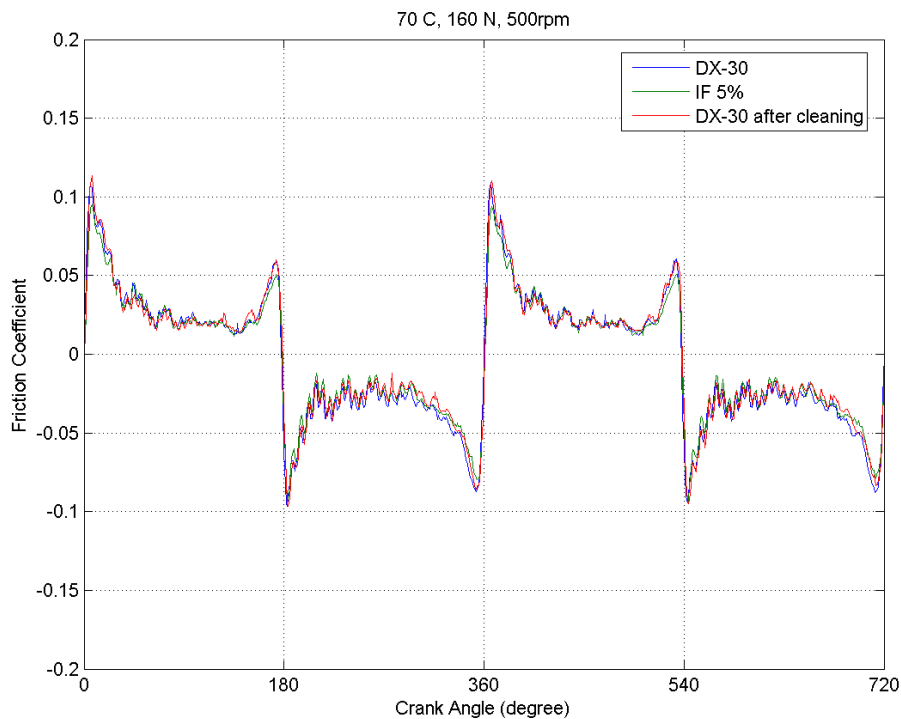


Figure 3.12: 5 % IF oil comparison results at 70 °C, 80 N and 300 rpm

Although the results given in Figure 3.12 showed that the IF formulated oil with this concentration has no effects on friction reduction, the results given in Figure 3.13 show a decrease when the load is increased to 320 N.

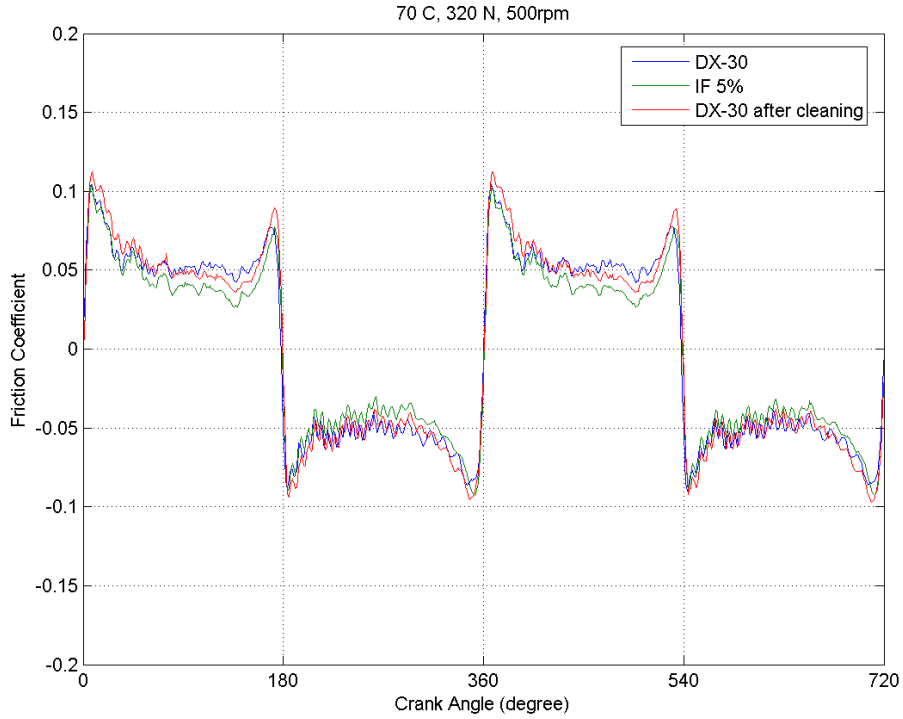


Figure 3.13: 5 % IF oil comparison results at 70 °C, 320 N and 500 rpm

Low temperature test results are given in Figure 3.14 and Figure 3.15. Similar to 1 % concentration results, IF nanoparticles have no effect in hydrodynamic regime.

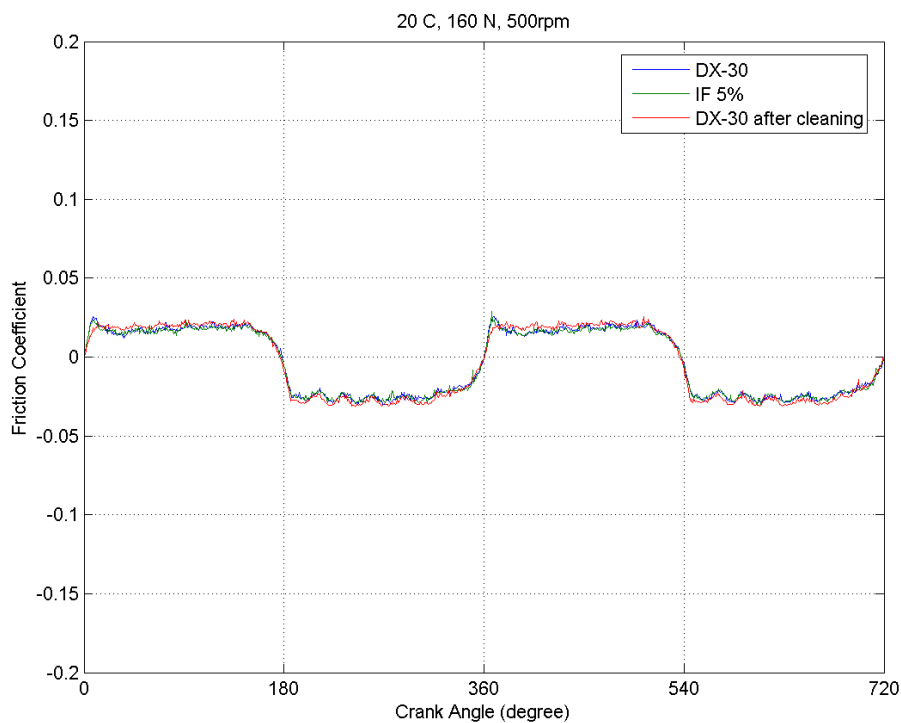


Figure 3.14: 5 % IF formulated oil comparison results at 20 °C, 160 N and 500 rpm

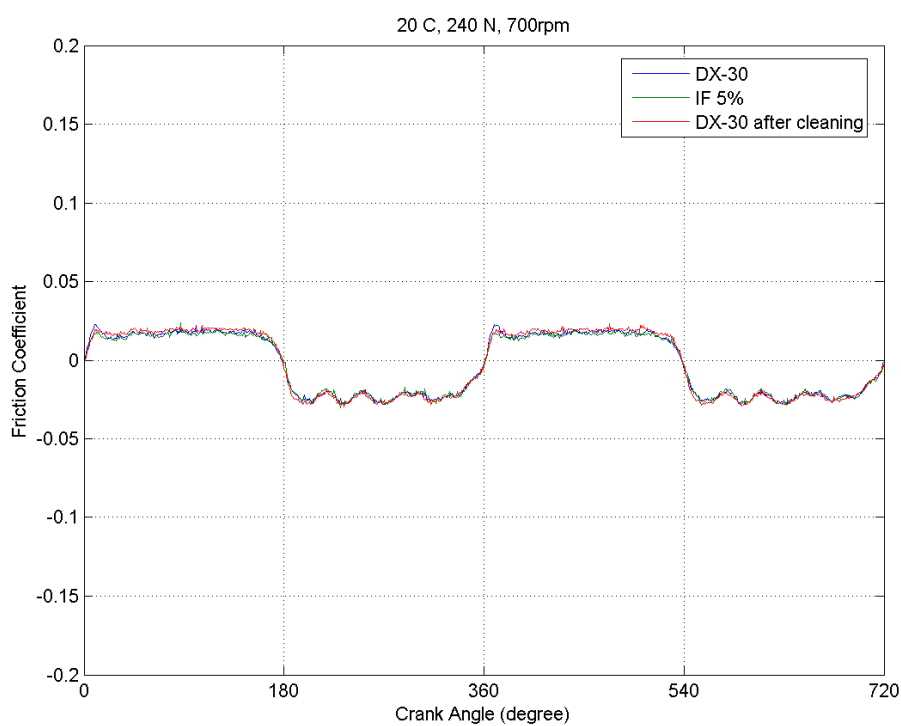


Figure 3.15: 5 % IF formulated oil comparison results at 20 °C, 240 N and 700 rpm

3.4 10 % IF-Oil Mixture Results

As the previous tests did not show a sufficient reduction in friction coefficient, the concentration is increased to 10 %, which is the most common use of IF nanoparticles in previous studies in the literature given in section one.

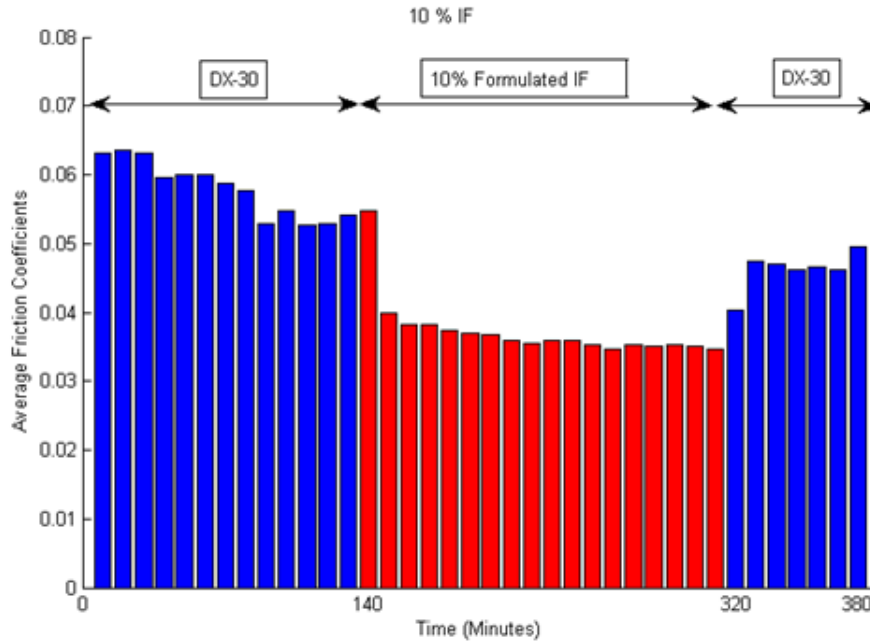


Figure 3.16: 10 % IF Experiment run-in results under 160 N with 500 rpm speed

In Figure 3.16, the running in tests of 10 % IF nano particle additive in DX-30 oil is shown. First part of the graphic shows run-in tests with reference which is also given with 1 % oil results. The second part is the 10 % IF concentrated oil run-in test. After the IF tests, base oil tests are repeated to examine the residual effects of the particles on the surface. Hexane solvent is used to clean the surfaces after each period. The running in tests are made at 160 N load and 500 rpm speed with 5 ml/h oil supply at 70 °C.

In this figure, 10 % oil tests shows more than 30 % reduction in average friction coefficient throughout 720 CA degrees collected by 10 minutes time intervals for 180 minutes. It is seen that the average friction coefficient is increased immediately for the first 10 minutes of IF operation. After that, a significant drop was observed and this drop continued gradually in one hour of operation.

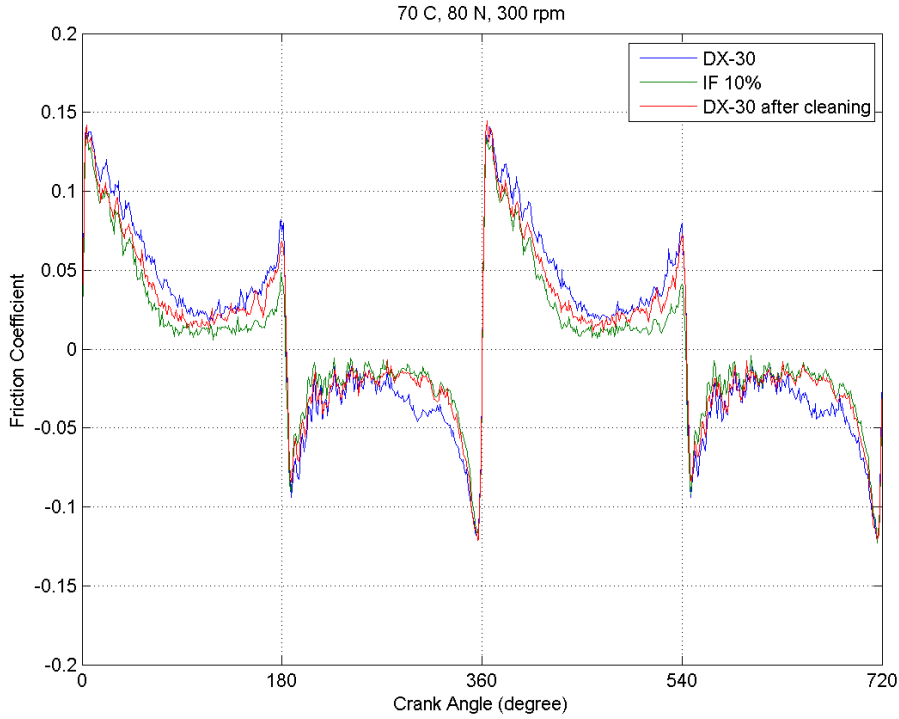


Figure 3.17: 10 % IF oil comparison results at 70 °C, 80 N and 300 rpm

In Figure 3.17, the results of IF nano particles with 10 % concentration at 80 N load and 300 rpm speed at 70 °C is compared with the reference oil test results. IF formulated oil results show a great decrease in friction coefficient for both mid-stroke and the dead centers. Also after cleaning the surfaces by hexane solvent, a partial influence can be also observed.

Keeping the speed constant, the load is increased to 320 N which is approximately 20 MPa of contact pressure given in Figure 3.18.

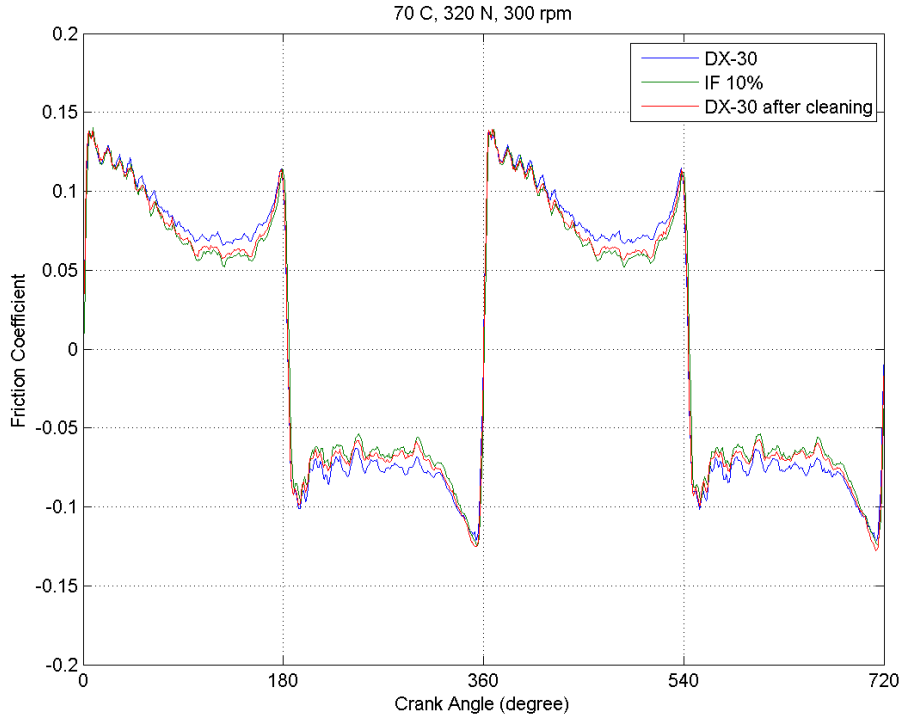


Figure 3.18: 10 % IF oil comparison results at 70 °C, 320 N and 300 rpm

As it seen from the Figure 3.19, the drop in friction coefficient still remains without supplying fresh IF particles into the contact region. The tribofilm does not disappear unless the lubrication conditions are boundary. The tribofilm at dead centers seems to be disappeared after stopping the IF oil supply. However, the mid stroke region still has lower friction coefficients compared to the reference oil test results. The power loss due to friction is proportional to the speed and the friction force. At dead centers, it is obvious that the speed is zero. The frictional power loss is negligible near dead center; nevertheless the mid-stroke friction coefficients are dominant in power losses, where the speed is higher.

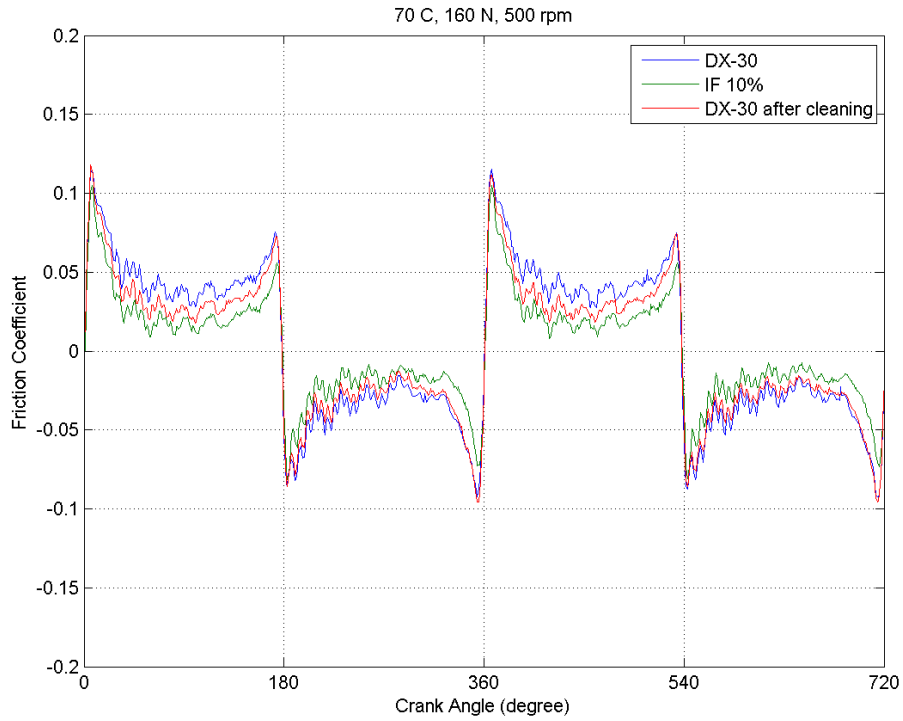


Figure 3.19: 10 % IF oil comparison results at 70 °C, 160 N and 500 rpm

The hydrodynamic lubrication regime tests at 20 °C with 10 % IF formulated oil is given in Figure 3.20.

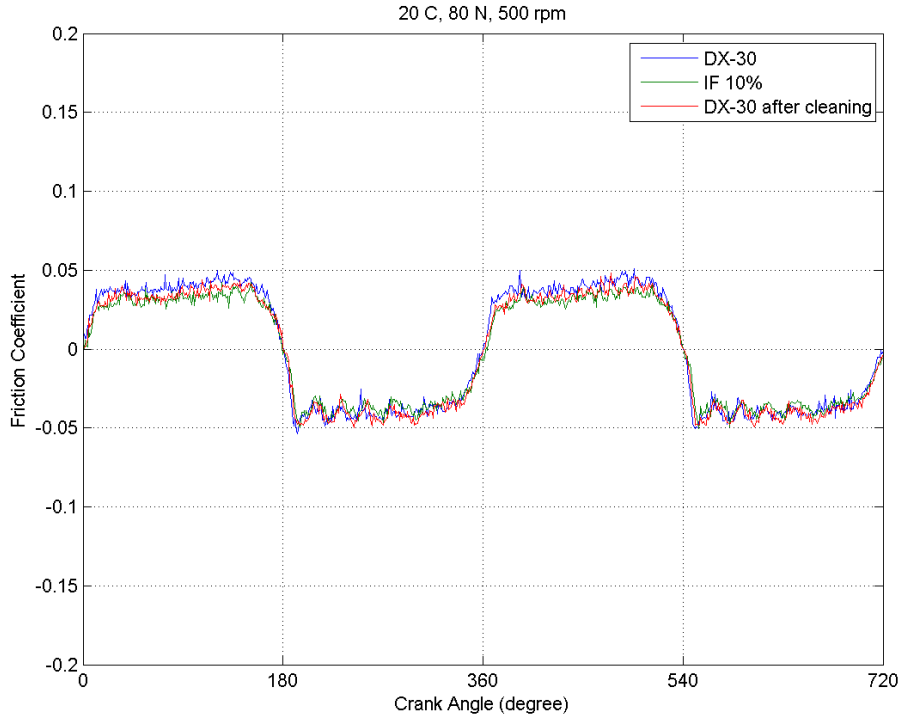


Figure 3.20: 10 % IF oil comparison results at 20 °C, 80 N and 500 rpm

3.5 5 % IF-Oil Mixture Results with Smoother Liner Surface

The previous tests did not show an influence in friction coefficient with low concentrations of IF-WS₂ nanoparticles added into mineral oil. It is because of the surface properties of the liner samples used for experiments. As mentioned, cylinder liners in internal combustion engines are plateau honed, so the surface roughness values are higher. In able to see the effects of surface roughness properties of the samples on tribological behavior of the nanoparticle additives, liner surface is smoothened with sand paper. Table 3.1 shows the original and the manipulated.

Table 3.1: Surface roughness values of the liner before and after sand papering

	Before	After
Ra	0.50	0.25
Rz	4.27	2.83
Rt	7.20	5.83
Rsk	-2.60	-4.00
Rp	0.97	0.53

Average surface roughness, R_a , is decreased from 0.5 to 0.25. The experiments are repeated with 5 % IF- WS_2 additive concentration. The running in results are given in Figure 3.21.

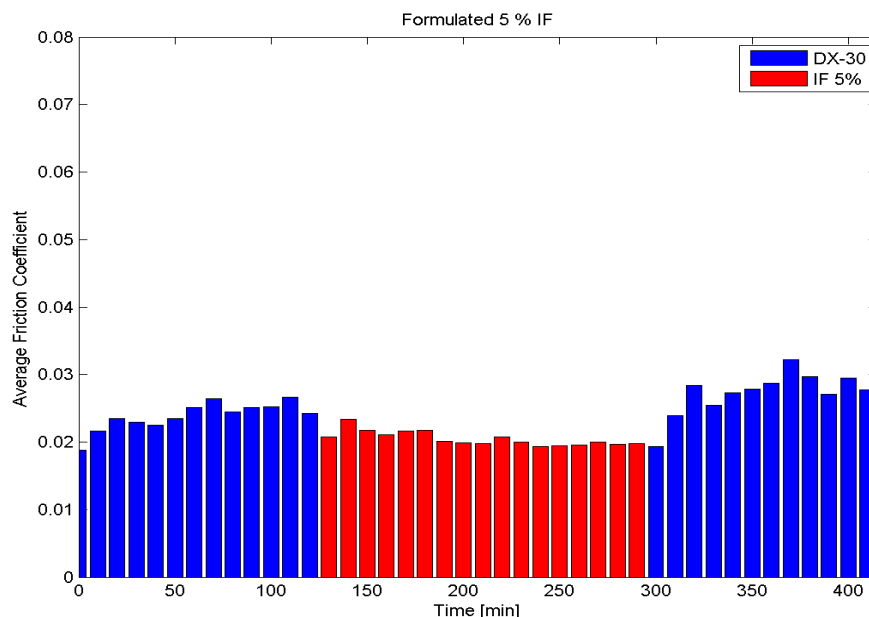


Figure 3.21: 5 % IF Experiment run-in results with modified surface

As the tests started with reference mineral oil DX-30, coefficient of friction seemed to be lower than the previous liner samples due to smoother surface due to higher tendency to develop hydrodynamic oil film. As the running in continued, friction coefficient started rising. During the running in period in previous tests, friction coefficient seemed to be falling. This contradiction is due to the modification of the surfaces.

With the use of 5 % WS_2 nanoparticle additive, a rapid improvement is observed and the friction coefficient stabilized to 0.02 values. As the surface roughness is reduced in this sample, the rolling effect of IF nanoparticles dominates the friction mechanism.

After nanoparticle added oil tests, reference oil is used for the last part of the running in operation. As it is seen from the Figure 3.21, the coefficient of friction gradually increased in the absence of nanoparticles in the contact region. Consequently, the surface started scoring. The picture of the surface is given in Figure 3.22.



Figure 3.22: Scoring of the liner sample

3.6 Raman Spectrum Analysis

After the tests, a detailed surface analysis is needed to determine the structure of the surface of both ring and liner segments. The inorganic fullerene nanoparticles in contact region is broken to individual sheets and these layers form a thin film with respect to the concentration, load, speed, etc., which dominates the reduction in friction coefficient [25-27]. The results of the Raman spectral analysis of the liner sample and the ring sample is given in Figure 3.21.

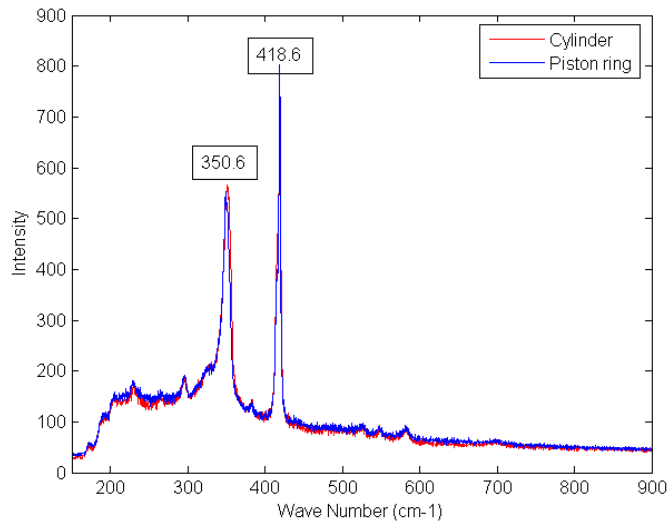


Figure 3.23: Raman Analysis of the Samples

As it is seen in the figure, the spectral results of both ring and the liner fit each other giving same peak values at same wave numbers. This means the same material is found on the surface of both samples.

Identification of the Raman spectra is made by comparing the results with the study of Virsek et al. in 2006. This study showed that WS_2 bulk or tube gives peaks approximately at 351 and 419 cm^{-1} . These values directly match the results of the Raman spectra of the ring and the liner samples.

The Raman analysis is repeated with different areas of the samples, but the shapes of the graphics and the places of the peak values did not change. These results show the presence of WS_2 film on the surfaces of tested samples. This film is the main reason of low friction values even after the repeated tests with reference mineral oil with cleaned surfaces.

4 CONCLUSIONS

The effect of IF-WS₂ nano-particles as an additive in engine oil was investigated in simulated engine conditions using a high speed and high stroke reciprocating friction simulator. This simulator has the ability to adjust speed, load and oil supply rate so that testing of different oils and samples in all lubrication regimes were available. During the tests, different concentrations of IF-WS₂ nanoparticles added into mineral oil was tested for a range of 0-20 MPa of contact pressure with 0-900 rpm speed at 20-70 °C of temperature. The following conclusions are formed based on experimental results;

- It is observed that low concentrations of IF-WS₂ nanoparticles in engine oil do not affect the friction coefficient in steady state operation. With the specific loads and speeds in engine applications, the concentration should be high enough in order to obtain satisfactory results.
- A tribofilm is formed on the contact surfaces after a short time of run, not at the same time when the IF formulated oil first met the surfaces. In fact, there is a rise in friction coefficient observed in first measurements, but after 10-20 minutes of operation, the influence is seen. This time is related with the concentration and the operating conditions. Thus, a running in is necessary to see the effects.
- The breaking of external sheets of IF-WS₂ spheres are seen to be dominant in friction reduction rather than the rolling and squeezing of these spheres. The sheets separate two surfaces from each other even in boundary lubrication region.
- The film forming rate is related with the fresh IF-WS₂ spheres on contact region, the speed and the load. This forming rate is important, since the sliding of the surfaces starts film removal process, and the balance of these processes determines the frictional characteristics.

- After the use of IF-WS₂ formulated oil, reference oil tests still show lower friction coefficients, meaning that the film formed on the surface is permanent at least in the mid stroke region where mixed lubrication properties are dominant. However, film thickness at the dead centers is too small that boundary lubrication regime is dominant and the effect of the film is diminished due to the removal of significant amount of the tribofilm.
- IF-nanoparticles are more effective where the surface roughness is lower. As the surface roughness value decreases to 0.25 values, scoring begins with the reference oil, and it is seen that IF-nanoparticles prevent scuffing. Smoother surfaces may be used in internal combustion engines that result lower friction coefficients with the use of IF-WS₂ nanoparticle additives in oil.

Further study is planned for a detailed dynamometer tests for final evaluation of IF-WS₂ nanoparticles in actual engine conditions. Before that, the test results of friction simulator system are planned to be used to see the effects of IF additives on fuel consumption. A model is being prepared for simulating Europe Duty Cycles in MATLAB environment for transmission of change in friction data to fuel consumption rate.

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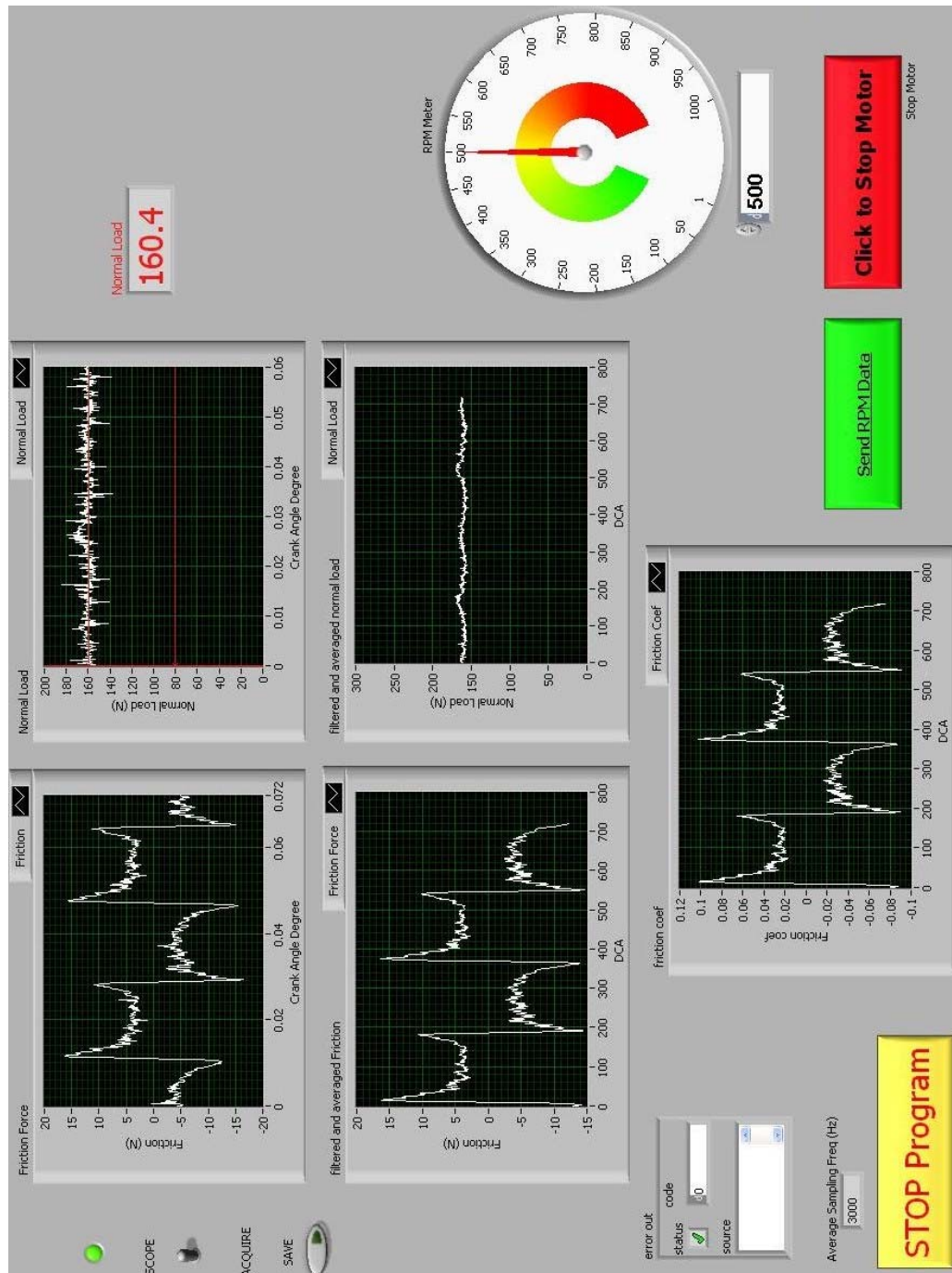
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APPENDICES

Appendix 1: User interface of LabView program



Appendix 2: Surface roughness values of the samples

		1%		5%		10%		Sand Papered	
		Before	After	Before	After	Before	After	Before	After
Right	Ra	0.59	0.64	0.50	0.47	0.49	0.48	0.25	0.30
	Rz	4.87	5.43	4.27	4.00	4.17	4.03	2.83	3.13
	Rt	8.73	9.63	7.20	6.27	6.47	6.53	5.83	8.03
	Rsk	-2.58	-2.39	-2.64	-2.24	-2.17	-2.26	-3.98	-4.84
	Rp	1.13	1.23	0.97	0.93	0.97	0.87	0.53	0.57
		Before	After	Before	After	Before	After	Before	After
Middle	Ra	NaN	0.55	0.43	0.44	NaN	0.46	0.29	0.24
	Rz	NaN	4.80	3.45	3.77	NaN	4.00	3.17	2.67
	Rt	NaN	9.67	5.55	6.70	NaN	7.57	6.77	5.23
	Rsk	NaN	-2.83	-2.02	-2.60	NaN	-2.60	-4.57	-3.81
	Rp	NaN	1.00	0.85	0.83	NaN	0.93	0.50	0.53
		Before	After	Before	After	Before	After	Before	After
Left	Ra	0.61	0.56	0.44	0.43	0.59	0.52	0.21	0.26
	Rz	5.03	4.80	3.73	3.50	5.00	4.30	2.23	3.10
	Rt	8.63	7.73	7.83	5.77	8.63	7.90	4.47	7.23
	Rsk	-2.07	-2.15	-2.89	-1.97	-2.19	-2.13	-3.72	-5.82
	Rp	1.17	1.13	0.83	0.83	1.13	1.07	0.43	0.57

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