

Developments and unsolved problems in nano-lubrication*

LUO Jianbin (雒建斌), LU Xinchun (路新春) and WEN Shizhu (温诗铸)

State Key Laboratory of Tribology, Tsinghua University, Beijing 100084, China

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Abstract The main achievements in the area of nano liquid film, e. g. the distinction between different lubrication regimes, properties of thin film lubrication, the transition between liquid and solid state, ordered and disordered state, the failure of thin lubricant film, the equivalent viscosity and flowing characteristics of micro-fluid, the influence of solid surfaces on nano-lubrication, thin film lubrication of polymer, superlubricity, have been reviewed and some unsolved problems are discussed.

Keywords: thin film lubrication, nano-tribology, lubrication failure.

1 The proposal of thin film lubrication

Since Reynolds suggested hydrodynamic lubrication theory in 1886^[1], boundary lubrication, elastohydrodynamic lubrication (EHL) and mixed lubrication have been proposed, which are very important for the perfection of lubrication theory. However, mixed lubrication just describes the friction characteristics when different kinds of lubrication regimes coexist. Thus, regardless of the film thickness or the tribological characteristics, there is an unknown region between EHL and boundary lubrication^[2]. Lubrication condition undergoes the following process with decreasing film thickness: (i) hydrodynamic lubrication, (ii) elastohydrodynamic lubrication, (iii) an unknown step, (iv) boundary lubrication, (v) dry friction.

Hydrodynamic lubrication and EHL have been studied thoroughly in both theoretical analysis and experiments. The boundary lubrication was proposed by Hardy^[3] in 1922. Afterward, several models^[4~7] were proposed in the 1950s. They contributed greatly to the theory of boundary lubrication. But how EHL transits to boundary lubrication and what properties of the transition region should be are still key problems in the lubrication theory^[8].

Usually, it is thought that when the thickness of lubricant film increases to a certain degree, the lubrication regime will change from boundary lubrication into mixed lubrication. In 1989, according to the demarcated range of friction coefficients and the film thickness, Luo and Yan^[2] found there is a blank between EHL and boundary lubrication, and this blank should be the transition region between the two regimes with both qualitative and quantitative changes. From 1988 to 1991, Spikes and

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Johnston^[9,10], using wedged spacer layer and optical interferometry to measure the changes of nanoscale lubricant film with influencing parameters, proposed the concept of super thin film lubrication. In 1992, the discussion was focused on sub-micro and nanoscale lubrication in Leeds-Lyon Symposium on Tribology. Some people named such lubrication ultra-thin film lubrication, while others named it fractionated thin film lubrication^[11]. In 1993, Wen^[12] and Tichy^[13] called it thin film lubrication. From 1994 to 1996, Luo and Wen et al.^[14~17], having developed a measuring equipment for nano-film thickness using relative optical interference intensity, investigated the lubrication characteristics and transition relation between EHL and thin film lubrication (TFL), and the time effect of TFL. They proposed a physical model for TFL, and defined the transition region between EHL and boundary lubrication as thin film lubrication. In later years, they also studied the failure of fluid film^[18], the influence of characteristics of solid surfaces on the film formation^[19], and proposed a new lubrication map to distinguish different lubrication regimes^[20]. In 1995, Tichy^[21,22], using the technique of numerical calculation, introduced directional viscosity problem and discussed the characteristics of thin film lubrication. In 1996, Hu et al.^[23,24], using the technique of molecular dynamic simulation, discussed the rheological characteristics and revealed the formation of ordered molecular structure. In 1999, Spikes et al.^[25] and Anghel et al.^[26] characterized the nano-film formed by different types of lubricants and additives, and Hu et al.^[27] studied the variation of effective viscosity of different molecular lengths using the surface force apparatus (SFA) and the molecular dynamic simulation.

2 The research areas and problems in thin film lubrication

The research of TFL was rapidly developed in 1990 s. It contributes greatly to the understanding of boundary lubrication and EHL, and brings many new related research areas, such as molecular ordered transition, the failure of thin liquid film, superlubrication, micro two-phase flow, confined fluid and origin of friction, etc.

2.1 Definition of thin film lubrication

Up to date, there are two view points about the definition of thin film lubrication. One is that thin film lubrication is an independent regime according to their lubrication mechanism^[16,18,19,23,27], and the other is that the lubrication in the nanoscale belongs to the extended boundary lubrication^[9,25,26].

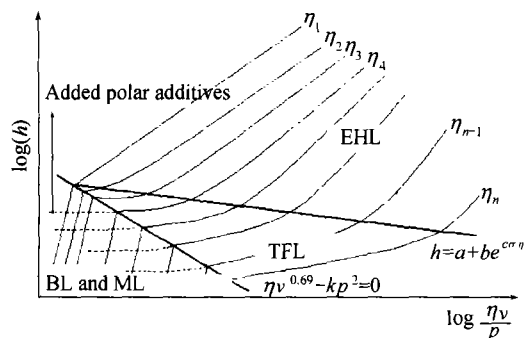


Fig. 1 Film characteristics in different regimes. Viscosity: $\eta_1 > \eta_2 > \dots > \eta_{n-1} > \eta_n$, $h/R_a > 3$.

Luo et al.^[20] proposed a relationship-diagram in terms of film thickness h , pressure p , speed v , lubricant viscosity η and the characteristics of additives and the solid surface as shown in Fig. 1. Fig. 1 shows the relationship between film thickness and its influencing factors in different regions with different lubrication regimes when the film thickness is three times larger than the combined surface roughness. In

the EHL region, the film thickness changes with speed by an exponential relation. The hydrodynamic effect becomes weaker as the film is thinner and the exponential index is no longer constant and the lubrication regime changes into TFL. The film thickness at the transition point is related to the lubricant viscosity η , solid surface tension σ , chemical characteristics of lubricant molecules, etc.; that is^[20]

$$h = a + b e^{c\sigma + k\eta}, \quad (1)$$

where a , b , c and k are constants related to molecular polarity, pressure viscosity property of lubricant, physical properties of solid surface, and h is the film thickness.

In TFL, when the speed decreases further, film will meet the failure point and collapses. When polar additives are added into the lubricants, the TFL regime will be maintained at lower speeds shown as the dotted lines in Fig. 1. The relation among the lubricant viscosity, rolling speed and pressure at failure points can be described as^[18]

$$\eta v^{0.69} - kp^2 = 0, \quad (2)$$

where η is lubricant viscosity in mm^2/s ; v speed in mm/s ; p pressure in MPa; k a coefficient which is 23.5×10^{-4} for lubricant without any additives. So pressure, speed and lubricant viscosity have great influence on the film failure. If the film fails, its thickness will suddenly decrease to a few nanometers.

Lubrication regimes should be divided into four types, i.e. HDL, EHL, TFL and BL. When the surface roughness is large enough as compared with the film thickness in the contact region, mixed lubrication will be formed. When the surface roughness is very small as compared with the film

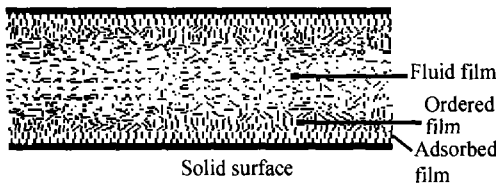


Fig. 2 Lubrication model.

thickness, a transition between lubrication regimes will take place according to the variation in film thickness as shown in Fig. 2^[15]. When the film is very thick, the fluid film plays a leading role and the lubrication property obeys EHL or DHL rules. As the film becomes thinner, the proportion of the ordered layer thickness to the total film thickness will become larger. When the

proportion is large enough, the ordered liquid layer will play a chief role and the lubrication regime changes into TFL. After the layer is crushed, the mono-molecular adsorbed layer will act as a chief role and the lubrication is boundary lubrication.

A method of using ratio h/R_a and h/R_g together to distinguish the lubrication regimes is shown in Fig. 3^[20], in which, h is film thickness, R_a the combined surfaces roughness and R_g the effective radius of lubricant molecules. The ratio h/R_g indicates the number of molecular layers in the gap between the two solid surfaces. When ratio h/R_a is larger than 3, three lubrication regimes

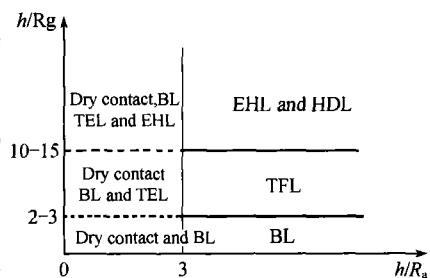


Fig. 3 Lubrication map. R_a , combined surface roughness; h , film thickness; R_g , effective molecular radius.

can be formed according to the ratio h/R_g . The regime is boundary lubrication when ratio h/R_g is less than 3, and it is TFL when ratio h/R_g is larger than 3 and lower than 10 to 15 which is related to the surface energy of the solid surfaces. It becomes EHL or HDL when ratio h/R_g is higher than 15. The mixed lubrication will be found when ratio h/R_a is smaller than 3. Different components of mixed lubrication with different values of the ratio h/R_g are shown in Fig. 3. Zhu and Hu^[28] studied the relation between film thickness and speed in the transition region between EHL and boundary lubrication. However, there are still some problems to be assured: (i) the definition of the transition between thin film lubrication and boundary lubrication; (ii) characteristics of mixed lubrication in the nanoscale; and (iii) the modification of lubricant molecules on the metal surface.

2.2 Characteristics of thin liquid film

In thin film lubrication, when film thickness is in the nanoscale, the effect of physical characteristics of solid surface on lubricant film becomes strong enough, especially for the surfaces with high energy, e.g. the surface of metal or metal oxides. In 1994, Dyakowski et al.^[29] analyzed the influence of solid surface tension on flowing characteristics of non-Newtonian fluid using numerical method. In the same year, Thompson et al.^[30], using molecular dynamic simulation, discussed the influence of solid wall on the action of lubricant molecules. Later on, Luo and Shen^[19,32] observed the influence of solid surface energy on the film thickness in the nanoscale in their experiment. A special property of thin film lubrication is the time effect on the film thickness in the nanoscale^[31,32]. As shown in Fig. 4^[31], the film thickness in the contact region hardly changes with time in the static state. At high speed, the initial film thickness is about 45 nm which is mainly in the hydrodynamic

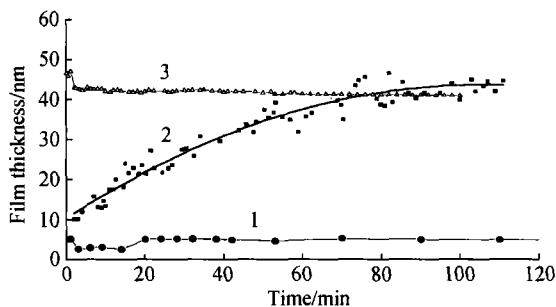


Fig. 4 Film thickness with time^[31]. 1, Decane + 3% palmitic, load: 4 N, $T = 30^\circ\text{C}$, $v = 0$ mm/s, ball: $\phi 25.4$ mm; 2, Decane + 3% palmitic, load: 4 N, $T = 30^\circ\text{C}$, $v = 3.12$ mm/s, ball: $\phi 25.4$ mm; 3, white oil No.1, load: 20 N, $T = 20^\circ\text{C}$, $v = 54.5$ mm/s, ball: $\phi 25.4$ mm.

state and the film thickness just slightly decreases with time. However, when the rolling speed is 3.14 mm/s, the film thickness obviously increases with time. The reason is known as follows: the ordered layer or solid-like layer near the solid surface becomes thicker in the running process.

When the number of molecules adsorbed on such layer and the number of the molecules washed out of the layer are equal to each other, the film thickness will hardly change with time. The thickness of nano-film is also related to the DC voltage applied. It increases with the voltage to a certain value and then hardly changes as shown in

Fig. 5^[32]. In addition, when the film thickness is in nano-scale, there is a great difference between the real viscosity and that of bulk fluid. Luo and Shen et al.^[15,32] observed that the measured film thickness is much thicker than the calculated result from Hamrock-Dowson formula^[33]. This indicates that the equivalent viscosity is much higher than that in EHL regime. Results from molecular dynamic simulation also show that the equivalent viscosity near the solid surface will become much higher than that of bulk fluid^[34,35]. Gao et al.^[36] also discussed the equivalent viscosity in the nano-film in experiment; however, there are some problems to be solved: (i) Why the equivalent viscosity of the

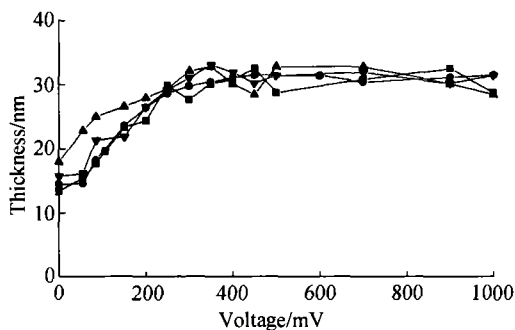


Fig. 5 Effect of DC voltage on lubricant film thickness^[32]. Load: 0.174 GPa; speed: 68 mm/s; temperature: 20 °C. ■, 2% CA + hexadecane; ●, 3% CA + hexadecane; ▲, 5% CA + hexadecane; ▼, 3% CAL + hexadecane.

film at nanoscale increases so much? (ii) To what extent do the surface effect of metal friction couple and the shear rheology affect the equivalent viscosity? What is the relation between them? (iii) In what degree do the size and polarity of lubricant molecules correlate with equivalent viscosity? (iv) What are the coexistence and transmission rules between fluid-like and solid-like state? (v) What is the mechanism of the influence of solid surface characteristics on the fluidity of micro-fluid? (vi) What is the upper limit of the equivalent viscosity of fluid film when it is close to the solid surface? (vii) How about the influence of the mechanical behaviour of solid surface and solid-fluid interface on the

lubrication characteristics? (viii) What is the correlation between friction and solid surface energy, roughness, hardness, crystal shape and size, electronic cloud density, etc.? (ix) What are the upper limits of the distance and the strength of the solid surface force which affects the liquid molecules nearby? (x) The stick-slip motion deserves more attention even though much work has been done on this area, but a scientific explanation is lacking.

2.3 The failure of thin film lubrication

With the progress in the research on fluid lubrication, the failure of liquid film has been observed in nanoscale. Such a phenomenon is closely related to the transition between thin film lubrication and boundary lubrication. As shown in Fig. 6^[18], as long as the pressure in the contact region is low enough or the lubricant viscosity is high enough, the hydrodynamic effect can be observed clearly even at very low speed. However, when the pressure increases to a certain value, a transition point can be observed in the speed-film thickness curve. As long as the speed reduces further, the film thickness will drop down rapidly to a few molecular layers and only the adsorbed or solid-like film is left. So the fluid film fails. The transition point is called the failure point which appears at different speed and pressure for different lubricants with different viscosities. The curve in Fig. 7^[18] is called the failure curve where the lubricants used are of alkyl without any polar additives. If the fluid factor $L (\eta_k v^{0.69})$ is in the region above the curve, the fluid film will be formed and the lubrication is in TFL or EHL regime. Whereas if it is in the region below the curve, the fluid film will collapse and the lubrication regime will change into the boundary lubrication or the mixed lubrication. Therefore, when the film thickness is more

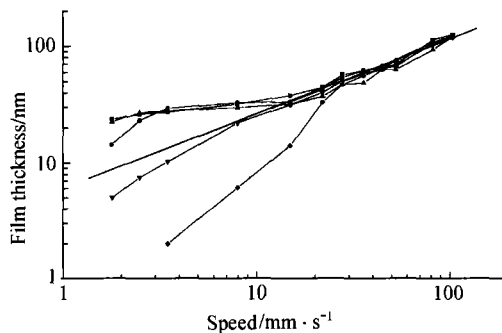


Fig. 6 Film thickness with speed^[20]. Temperature: 28 °C, Pressure: 0.185 GPa. ◆, C₈H₁₈; ▲, C₁₀H₂₂; ●, C₁₂H₂₆; ■, C₁₆H₃₄.

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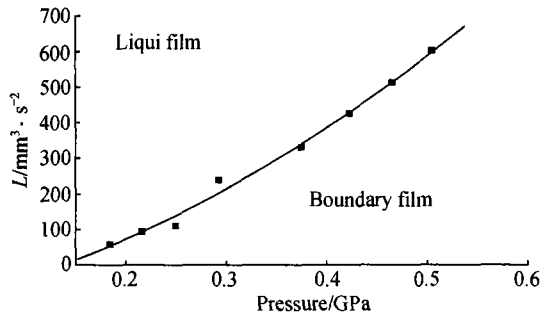


Fig. 7 Failure of liquid film^[18].

than three times of surface roughness R_a , the relation between failure fluid factor L_f and failure pressure P_f , at which the failure of the fluid film takes place, is

$$L_f = 23.5 \times 10^{-4} P_f^2, \quad (3)$$

$$L = \eta_k v^{0.69}, \quad (4)$$

where η_k is the kinetic viscosity of lubricant in mm^2/s ; v the speed in mm/s . The unit of P_f is

MPa and that of L_f is mm^3/s^2 . If the two surfaces need to be lubricated with the fluid film within the TFL and EHL regime, the lubricant and the rolling speed should be chosen according to the pressure applied as calculated from Eq. (3), so as to make the liquid factor L larger than the failure fluid factor L_f . Otherwise, the liquid film cannot be maintained under such a pressure. The following problems need to be studied: (i) The physical mechanism of film failure; (ii) the failure of fluid continuum condition; (iii) after the fluid continuum condition fails, how to describe the movement of lubricant molecules?

2.4 Solid-liquid transition and ordered molecular film

Two of the key problems in the research of thin film lubrication are in what state, e.g. solid, glassy, ordered liquid or general liquid, the layer will be when it is in the nanoscale and under what condition the solid-liquid phase change will take place. These problems are related to the characteristics of solid surface and the structure of molecules, e.g. spheric, chained or dendritic.

The ordered molecular film has the characteristics of stable performance, controllable thickness, high coherence with substrates^[37]. There are six kinds of ordered molecular films which are useful in the tribology, i. e. Langmuir-Blodgett (L-B) film^[38], self-assembly film^[39], molecular deposition film^[40], film by molecular epitaxial growth^[41], high oriented solid ordered film^[42], and shear induced ordinary film^[43]. L-B technique is the earliest one and has been widely used. Nevertheless, the adhesion between LB film and the substrate surface is still to be improved. Rapidly developed self-assembly film is formed by the polar molecules owing to the chemical adsorption. At present, single lipase layer or other organic film can be made on the surface of Au, Ag, Cu, Al_2O_3 , glass, and so on. However, it requires further research on thickness control, performance stability and the match between molecular structure and substrate. Epitaxial growth method, formerly used in the research of crystal growth, was used in the research of organic molecular growth on the crystal surface^[41] and the work is just at preliminary stage. Solid ordered films, such as MoS_2 and highly oriented pyrolytic graphite (HOPG)^[42], forming ordered slippery layer during movement, not only reduce friction coefficient greatly, but also have good stability and anti-compressive ability. Their main shortcoming is the difficulty in controlling the film thickness in the nanoscale. The shear induced ordered film is the film that the disordered fluid lubrication molecules turn into ordered arrangement under the effect of surface energy, shear induction or external field^[31,37]. Such a film

results in the reduction of friction coefficient^[44-46]. In the research of superlubricity^[47,48], more attention has been paid to such an area.

In summary, research on characteristics of ordered molecular film is undergoing a rapid development, and different types of ordered films have their own advantages and shortcomings. Even though many experimental and numerical calculated results have been obtained, the following problems also remain to be clarified: (i) What factors and to what extent do they influence the molecular ordering? (ii) What are the relationships between the degree of molecular order and equivalent viscosity or friction force? (iii) What is the thickness limit of the ordered layer caused by the solid surface force?

2.5 Thin film of polymer

The research on characteristics of polymer develops rapidly because polymer has large molecular weight, good flexibility, high cohesive strength with solid surface^[49-51]. Klein et al.^[52,53] investigated the lubrication properties of polymer film by a surface force microscope (SFA) and proposed that polymer lubricant has two characteristics: (i) under the effect of double electric layer and dispersion force, a repulsive force field is setup between the two solid surfaces. The force field is related to polymer structure, polarity, flexibility, and solvent. When chosen suitably, a repel field will be formed which is 6 to 15 times greater than the diameter of molecule group, i. e. several hundreds of nanometers. (ii) The polar end of polymer film can combine with solid surface and form a strong surface film by implanting into the solid surface or by adsorption. Molecules may stretch, but they will not separate from solid surface. The tribological properties of polystyrene-polyethyleneoxide (PS-PEO) which can graft into the solid surface was investigated by Shi et al.^[51]. However, polymer is very complicate in structure and has much different properties with different groups. Here are the problems need to be solved: (i) How to use polymer as lubricant of superlubricity? (ii) What is the lubrication mechanism of different groups of polymer? (iii) What is the influencing mechanism between the structural characteristics of polymer and the surfaces of friction couple? (iv) How to set up a repulsive force field in the contact region using molecular properties?

2.6 Superlubricity

The appearance of superlubricity^[54,55] has attracted the attention of researchers in the fields of tribology, machinery, physics and chemistry. Theoretically, superlubricity is the realization of zero friction condition. But in practice, it is thought that when friction coefficient is in a scale lower than 0.001, the lubrication condition is in superlubricity^[56]. The research on superlubricity and its mechanism will be very helpful to understand lubrication and origin of friction.

At the beginning of the 1990 s, by theoretical calculation, Hirano and Shinjo^[56] found that when two arranged crystal surfaces move in certain commensurate surfaces and directions, friction vanishes, or superlubricity state takes place due to the weak mutual interaction and relaxation between molecules. However, their experimental results^[54] of superlubricity have not been accepted because the measurement precision was not high enough. Preliminary progress has been made in the investigation of the mechanism of superlubricity and the condition of existence both in experiment^[54]

and theory^[55]. There are two kinds of materials that have superlubricity-like properties. One is solid lubricants, such as high oriented pyrolytic graphite and MoS₂ which show an ultra-low friction in the special direction under high vacuum condition. Another kind is polymer materials with end-grafted chains^[52], e.g. PS-PEO. Klein et al.^[53] and Yoshizawa et al.^[47] did some experiments on the end-grafted chain molecular film using a surface force microscope. The 'molecular brush' layer is formed on the mica surface. A repulsive force is produced in the range of $0 \sim 2L$ (L is the length of molecular chain), and it increases rapidly with a decrease in the distance between two mica surfaces. Under the effect of repulsive force, two mica surfaces are separated and can slide freely, so an ultra-low coefficient about 0.001 can be achieved at room temperature.

In summary, superlubricity research is at its preliminary stage. A systematic theoretical analysis of the mechanism of superlubricity is needed^[59]. In addition, the ordination of lubricant molecules is required to realize superlubricity at room temperature. Random fluid molecules not only dissipates energy due to entanglement and collision during shear process, but also cannot establish stable repulsive field. The following problems should be solved in this area. (i) What is the relationship between molecular ordered degree and superlubricity? (ii) What is the mechanism of superlubricity? (iii) What are the basic conditions for the transition from non-superlubricity state to superlubricity state? (iv) What are the physical and chemical characteristics that lubricant molecules should possess to achieve superlubricity state?

2.7 Origin of friction

For tribologists, the origin of friction is always a baffling problem. Though friction force belongs to one of the basic four forces in physics, and there are molecular theory, mechanical theory and molecule-mechanical theory of friction, but the understanding of the origin of friction is still not adequate.

Atom force microscope (AFM)^[60] and friction force microscope (FFM) have been widely used in surface science and nano-tribology researches^[61] since they were invented. Many friction phenomena at atomic scale are discovered. Ruan and Bhushan^[62] showed that surface adhesion energy and force directly account for contact deformation and adhesion, and some material may contact and deform because of interfacial interaction even under zero load; micro-friction force is related to contact area and ultimate adhesion shear stress, which is much lower than macro-friction force; micro-friction force has correlation with 2D surface topography, and both have the same wave period. This makes possible for controlling friction by changing micro-topography and adhesion force. Further, stick-slip, the alternate occurrence of static and dynamic friction, is an integrated characteristic of sliding friction which is responsible for instability. Yoshizawa et al.^[63] found that the stick-slip is caused by interfacial phase transition and adhesion energy changes; with the increase of sliding speed, amplitude of friction decreases, frequency increases and stick-slip disappears at a certain sliding speed; single molecular boundary film of some materials have performed super dynamic friction with even lower friction force under high speed. Experiments also show that energy consumption in boundary lubrication correlates with visco-elastic behavior of lubricant film and with viscous lagging. They have the same mechanism at molecular scale. The following problems are crucial: (i) What

kind of intermolecular force does friction belong to? (ii) What kind of the friction force is related to the physical and chemical properties of friction couple, and what is the relationship? (iii) What kind of factors affects friction behaviour at atomic scale?

2.8 Two-phase flow

There are impurity grains in almost all the friction process, especially in micro/nano tribology. The size of impurity grains is in the same scale with the lubrication gap, and therefore their influence is very important, especially on wear. It is difficult to control the size, shape and movement of the impurity particles; therefore the research concerning impurity has lagged behind other researches in micro-tribology. Particle has two characteristics, i.e. mobility and heterogeneity. Mobility means that it appears and acts at any time in any friction process, and it cannot be protected thoroughly. Heterogeneity means that the impurity grains have various chemical compositions, different kinds of metal or organic grains. These factors make the research of it difficult and complicated. Patton and Bushan^[64] discussed the effect of fine particles on the meniscus force. Shen et al. observed the influence of nano-particles of diamond added into base oil on the film thickness and friction properties in nanoscale (unpublished data). Although certain progress has been made on single impurity grain of magnetic recording system^[65-67], some problems remain to be solved. (i) The effect of grain geometrical parameters under different working conditions; (ii) the transportation mechanism of grains; (iii) perfection of the real time observing technique for grain movement.

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