

# Lubrication Behavior of Nano SiO<sub>2</sub> Cluster in Water-based Cold rolling Liquid

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**Abstract.** As a kind of new lubricant additive, Nano SiO<sub>2</sub> particles exist in the form of particle cluster in suspensions. The objective of the present study is to put forward the lubrication behavior of Nano SiO<sub>2</sub> clusters in water-based cold rolling liquid through cold rolling experiments of Q235 steel. PH values were adjusted to find out the best degree of dispersion. Samples were investigated through micro surface observation and elemental analysis. It is found that Nano particle clusters strengthened the spreading property of lubricant film and enhanced the stability by splitting into smaller clusters under rapid increased stress in micro bulge zone. Those split clusters reshaped into larger clusters again though the attractive force between micro particles, thus actions became circulations. This study focus on the behavior of nanoparticle clusters in lubricating instead of each single Nano fines, which is more practical and applicable.

## Introduction

Nanoparticles, as a kind of new lubricant additive which possess properties such as quantum-size effect, the small size effect, and surface effect[1], have aroused marked attentions because of their special behaviors in wear resistance, load carrying capacity and anti-friction ability[2,3,4]. Nano SiO<sub>2</sub> is one of those typical fines which has controllable particle size, nontoxic property, and extreme pressure[5,6,7]. Aggregation of nanoparticles causes poor surface quality and increase of roughness, being as abrasive instead of lubricant additive[8]. So lubricating properties of Nano lubricant depends on the dispersion of additive particles a lot[9], with obviously effect on the quality of friction surface.

The lubrication behavior of nanoparticles has been researching since the birth of Nano tribology. Recent research found that lubricating actions were closely related to the characteristic features of deposition film formed on the contact friction pair by chemical reactions[10]. F. Honda[11] found that nanometer thick Ag layers could resist shearing while lubricating and exhibit anisotropic performance within the thickness range of nanometers. As for the micro smooth surface, nanoparticles could transform sliding friction into rolling friction, which requires the narrow range of size distribution, superb condition of dispersion and high rigidity[12]. In order to find out the practical lubricating behavior in real working condition, this work presents investigations of lubrication behavior of Nano SiO<sub>2</sub>, regarding Nano-scale nanoparticle clusters as a whole carrying load while lubricating which is not only theoretical but also practical.

## Experimental Details

### Solution Preparation

The analyzed purity ethanol and deionized water were used as base fluid, silica fines with analyzed purity of 99.8% and mean diameter of 15nm were used as lubricant additive. Using electronic balance to weight 0.3g silica fines and 2g water-soluble borate, added them into the base fluid which contained 80ml deionized water and 20ml ethanol. The volume of liquid was measured by the graduate which was cleaned after each procedure using deionized water and dried. Adjusted the pH values to reach the best dispersion, chemical reagent were diluted hydrochloride and calcium

carbonate. Stir the suspension for 5 minutes and then ultrasonic it for 10 minutes. The degree of dispersion was analyzed using a Malven Instrument Ltd WR-16IAQ zeta meter.

### Lubrication Experiment

Cold rolling experiment of Q235 steel was researched using the 4-Hi reversing mill. Two samples were rolled and textured, one was with cold rolling liquid contained Nano SiO<sub>2</sub> as lubricant, the other used the base fluid as lubricant only, textured as the controls. The roughness measurement and micro surface observation were researched using Olympus OLS400 laser scanning confocal microscope and D3100&NanoscopeV atomic force microscope. Elemental composition of micro surface was textured using Quantum 2000 XPS. And the analyzed surface (100μm~100μm) was etched 1nm by the sputtering argon ions to minimize the experimental error. Binding energy C1s=284.8eV was used as correction. Strips used in the experiment were with the dimensions of a thickness of 2.3mm, a length of 15cm and a width of 10cm. The roll was cleaned by acetone.

## Results and Discussions

### Solution Preparation

Six samples were set to find out the best degree of dispersion in room temperature, and the only variable was pH values in this group of samples. The pH values of samples were showed in table I.

Table I. PH value of the suspension in room temperature

Number	1	2	3	4	5	6
pH value	2.9	5.4	6.7	9.4	10.7	12.1

Considering the high surface energy and micro dimension of nanoparticles, aggregation happens when they are added into base fluid and dispersed. Fig.1 shows the trend between relative size of cluster in the suspensions and pH values.

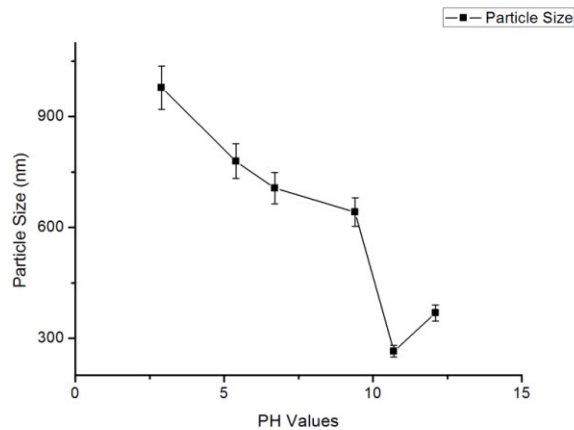


Fig.1 Relative size of particle cluster in suspensions with different pH values

With the rise of pH value, the size of solid spheres decreases until it reaches 264.8nm when pH is 10.7, then begins to increase. Considering the mean diameter of Nano-silica fines(15nm), those nanoparticles existed in the form of clusters in the cold rolling liquid. And the larger the mean diameter tested are, the stronger the aggregation is, the worse the dispersion is. Best degree of dispersion happens when pH value is 10.7.

## Lubrication Experiment

The surface roughness of the roll was measured and taken the average as  $Ra=0.2\mu\text{m}$  before the experiment. Fig.2 (a) (b) shows the 2D micro surface of rolled Q235 steel.

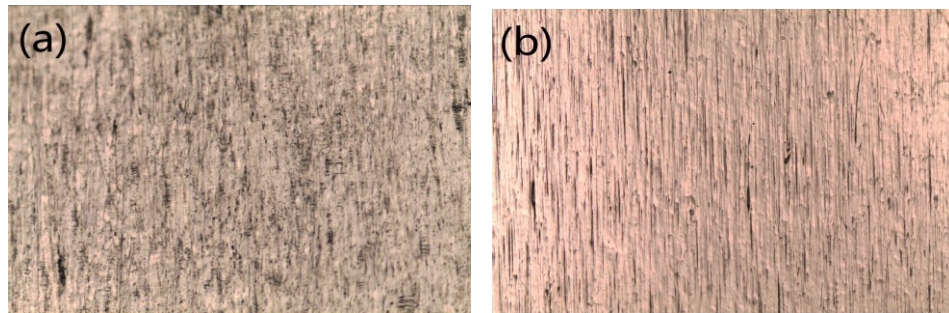


Fig.2 Micro surface (2D) of cold rolling Q235 steel, (a) pH=10.7 base fluid as lubricant, (b) pH=10.7 base fluid containing Nano  $\text{SiO}_2$  as lubricant

Defects such as spot and plowing can be seen on the micro surface of samples with pH=10.7 base fluids as lubricant, distribute randomly. And the roughness of surface is 0.22, which is close to the roughness of roll.

Compare sample (a) with (b), we can find that the distribution of spot and plowing is similar, although the quality of micro surface of sample (a) are poorer, more spots and plowings can be seen, for the base fluid without Nano  $\text{SiO}_2$  hardly played as a role of lubricant while rolling. Table II shows the extreme pressure of cold rolling liquids with different pH values. Dispersed Nano-silica in suspension increased the viscosity and extreme pressure of lubricant, provided a strong support during cold rolling. Such property leads to a better quality of micro surface, and the roughness of sample (b) is  $0.15\mu\text{m}$ , which is lower than the average roughness of the roll.

Table II. Extreme pressure of the suspension with different pH values

pH value	2.9	5.4	6.7	9.4	10.7	12.1
$P_B$ (N)	259	436	510	558	624	578

Furthermore, we analyzed the 3D micro surface of defects and normal parts taken from the sample showed in Fig.2 (b). Typical defects are showed in Fig.3 (a) and typical normal part is showed in Fig. 3 (b). Majority of spots distributed on the direction of metal flow, while most plowing distributed vertically to it. Spots and plowings from same area coverage can be much more different about depth and micro acreage. The intrinsic changes of grain size can be taken as a mainly factor, grains grow in the direction of metal flow, causing defects' growth too. Furthermore, the interaction between cold rolling liquid and samples matters a lot too.

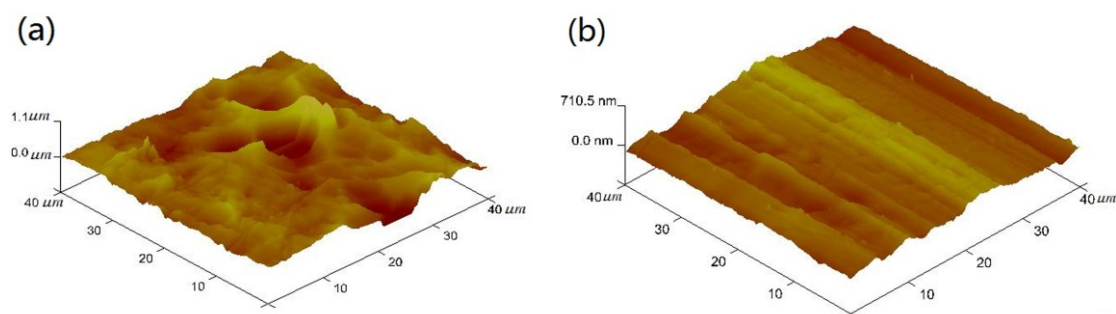


Fig.3 Micro surface (3D) of cold rolling Q235 steel, pH=10.7 base fluid containing Nano-silica as lubricant

In order to observe the lubricating behavior of Nano SiO<sub>2</sub>, the elemental composition of defect and normal surface (Fig.3) were analyzed. The content of Si element is about 3.12% on the normal surface, while 2.01% Si element can be found on the defect surface. The narrow band spectra scanning results of Si element is showed in Fig.4, which agrees with that more Si element remains on the normal surface after the experiment.

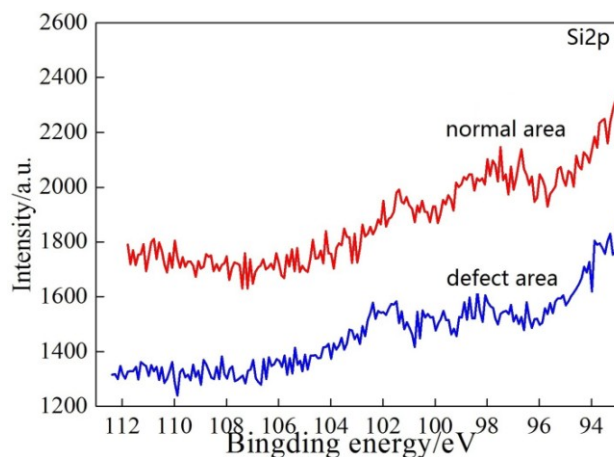


Fig.4 Narrow band spectra scanning results of Si element

Compare with the elemental composition of Q235 steel which is showed in table III, the content of Si element is much higher, whether on the defect surface or on the normal surface.

Table III. Elemental composition of Q235 steel

Element	C	Mn	Si	S	P
Content	0.12%~0.20%	0.30%~0.670%	≤0.30%	≤0.045%	≤0.045%

It is possible to infer one conclusion from this set of facts that Si element comes from the lubricant suspension, which remains on the surface of samples after rolling. The Nano SiO<sub>2</sub>, as lubricant additive, moved with the lubricant which had the same direction of motion as metal flow while rolling, filled in the plowings and spots. Some of the spots were filled by the Nano SiO<sub>2</sub>, thus roughness of surface decreased. Some plowings were filled partly and looked like spots too, thus the distribution of spots was along with the direction of motion of metal flow.

The Nano SiO<sub>2</sub> existed in the form of Nano particle cluster in suspension while rolling. This Nano particle cluster distributed in lubricant evenly and enhanced the wettability of suspension, which enhanced the spreading property of lubricant film. Fig.5 shows the wettability of lubricant without or with Nano SiO<sub>2</sub> as additive. The wetting angle decreased with the adding of Nano SiO<sub>2</sub>, and the wettability increased.

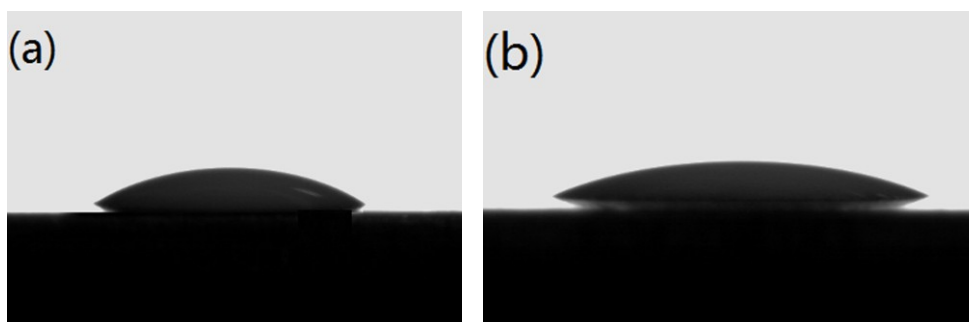


Fig.5 The wettability of lubricant without (a),  $\theta=33.6^\circ$ ; and with Nano SiO<sub>2</sub> (b),  $\theta=18.4^\circ$ , as additive

The process of present experiment and lubricant behavior of Nano SiO<sub>2</sub> in rolling deformation zone are shown in Fig.6. The liquid suspension itself plays an essential role in anti-friction ability, and the film of lubricant has reduced the wear such as adhesive and abrasive. Owing to the roughness of micro surface, the force of deformation zone carrying by lubricant can be complex: a higher stress exists near the micro bulge (square A in Fig.6). Meanwhile, the micro bulge of roll will cause the spots and plowing on the surface of samples (square B in Fig.6).

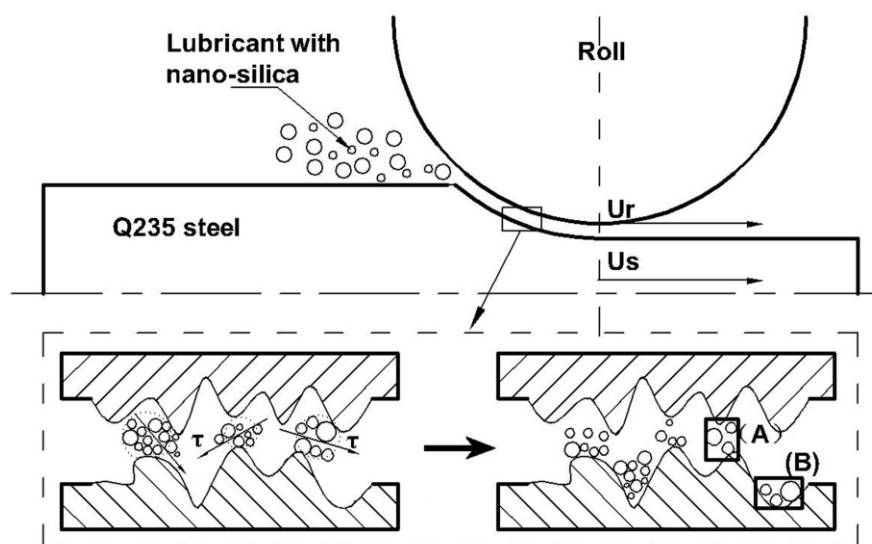


Fig.6 Schematic diagram of lubricant behavior of Nano SiO<sub>2</sub> in rolling deformation zone

When moved near the micro bulge zone, the Nano particle cluster split because of the rapid increase of stress and the contact with micro bulge. The mean dimension of Nano particle cluster became smaller under the shear stress, as shown in Fig.6, this action increase the stability of lubricant film under high stress. Some of those smaller particle clusters remained near the micro bulge zone, increased the extreme pressure property of lubricant and prevented the lubricant failure caused by film fracture (square A in Fig.6). Some of those smaller particle clusters filled into spots and plowings near the micro bulge zone under the high stress and renovated the defects (square B in Fig.6). With the movement of lubricant, suspended small particle clusters will reshape into larger clusters owing to the attractive force between micro particles, and the action became circulation.

## Summary

Nano SiO<sub>2</sub> particles exist in the form of clusters in the cold rolling liquid. Best degree of dispersion happens when pH value is 10.7.

Nano SiO<sub>2</sub> clusters play a role as lubricant additive which strengthened the spreading property of lubricant film and enhanced the stability near micro bulge zone.

Dispersed Nano SiO<sub>2</sub> clusters split into smaller clusters near micro bulge zone under rapid increased stress and reshape again because of the attractive force between micro particles while lubricating, those actions become circulations. Smaller clusters refill the defects and bigger clusters carry loads near micro bulge zones.

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