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## Effects of oil-in-water based nanolubricant containing TiO<sub>2</sub> nanoparticles in hot rolling of 304 stainless steel

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# Effects of oil-in-water based nanolubricant containing TiO<sub>2</sub> nanoparticles in hot rolling of 304 stainless steel

## Abstract

Energy saving and improvement of product quality are of crucial importance in hot rolling of 304 stainless steel. In this paper, oil-in-water (O/W) based nanolubricants containing TiO<sub>2</sub> nanoparticles were developed to reduce the rolling force and improve the surface quality of rolled 304 stainless steel product. Practical hot rolling tests with and without application of lubricant were conducted to systematically investigate the effects of the developed O/W based nanolubricants on the rolling force, surface roughness, oxide scale thickness and tribological behaviour. The obtained results indicate that the nanoparticles can enter the deform zone with oil droplets to take a lubrication effect. The optimal lubrication effect can be achieved when the O/W (1% oil mass fraction) based nanolubricant with a TiO<sub>2</sub> mass fraction of 1.5% was applied. The novel nanolubricant has a great potential to be applied in the hot steel rolling, to realise the cost-effective and environmental-friendly manufacturing process.

## Keywords

stainless, 304, rolling, hot, nanoparticles, effects, tio<sub>2</sub>, steel, containing, nanolubricant, oil-in-water

## Disciplines

Engineering | Science and Technology Studies

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## Effects of oil-in-water based nanolubricant containing TiO<sub>2</sub> nanoparticles in hot rolling of 304 stainless steel

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### Abstract

Energy saving and improvement of product quality are of crucial importance in hot rolling of 304 stainless steel. In this paper, oil-in-water (O/W) based nanolubricants containing TiO<sub>2</sub> nanoparticles were developed to reduce the rolling force and improve the surface quality of rolled 304 stainless steel product. Practical hot rolling tests with and without application of lubricant were conducted to systematically investigate the effects of the developed O/W based nanolubricants on the rolling force, surface roughness, oxide scale thickness and tribological behaviour. The obtained results indicate that the nanoparticles can enter the deform zone with oil droplets to take a lubrication effect. The optimal lubrication effect can be achieved when the O/W (1% oil mass fraction) based nanolubricant with a TiO<sub>2</sub> mass fraction of 1.5% was applied. The novel nanolubricant has a great potential to be applied in the hot steel rolling, to realise the cost-effective and environmental-friendly manufacturing process.

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**Keywords:** Nanolubricant, TiO<sub>2</sub> nanoparticles, Oil-in-water, Hot rolling, Stainless steel;

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## 1. Introduction

Most of metals were primarily produced by rolling in metalworking processes [1]. Reduction of rolling force and roll wear, energy saving and improvement of product quality are still challenge in modern rolling industry. In recent years, oil-in-water (O/W) emulsion has been in use in industrial rolling mills to achieve these objectives [2], due to their good cooling and lubrication.

In order to improve the efficiency of lubrication further, solid additives, including nanoparticles, in oil-based lubricant have been investigated by many researchers [3]. The oil lubricant containing nanoparticles can be called nanofluid/nanolubricant. A nanofluid is a fluid (e.g. water, ethylene glycol and oil) with dispersed metallic or non-metallic nanoparticles with a typical size of less than 100 nm. As one branch of nanofluid, research on nanolubricant has been attracting researchers' interest. Few researchers, however, have been focused on the investigation of O/W based nanolubricants.

In our research, O/W based nanolubricants containing TiO<sub>2</sub> nanoparticles were developed and effects of the nanolubricants in hot rolling of 304 stainless steel were investigated.

## 2. Experimental procedure

O/W based nanolubricants with a different mass fractions of TiO<sub>2</sub> nanoparticles ( $\varphi=0, 0.5\%, 1\%, 1.5\%, 2\%, 4\%$ , and 6%) were prepared by the treatment of high-speed stirring and ultrasonic. They were named O/W, 0.5TiO<sub>2</sub>, 1TiO<sub>2</sub>, 1.5TiO<sub>2</sub>, 2TiO<sub>2</sub>, 4TiO<sub>2</sub> and 6TiO<sub>2</sub>, respectively. Rutile TiO<sub>2</sub> nanoparticles with a sphere-like particle size of 30 nm and a lubricant oil with a viscosity of 1.20 Pa·s and a density of 0.90 g/cm<sup>3</sup> at 23 °C were used. The lubricant oil can be as a carrier of nanoparticles and a resource of the oil film in the contact area during hot rolling process. In this procedure the nanoparticles were mixed with the distilled water first using high-speed stirring mechanical mixture for 3 min. Then, the lubricant oil (1%) was dropped into the TiO<sub>2</sub>/water fluid under high-speed stirring for 3 min. Finally, the prepared nanolubricants were treated in an ultrasound bath for 10 min. The mechanical stirrer and ultrasonic processor were used to ensure good dispersion of the nanoparticles and homogeneous of the nanolubricants. In this way, the nanolubricants with good dispersibility (average TiO<sub>2</sub> nanoparticle agglomerate size was 290 - 420 nm) and stability (the TiO<sub>2</sub> nanoparticle relative fraction after 72 h were maintained over 80%) were prepared.

304 stainless steel was employed in this study. Its chemical compositions are shown in Table 1. The machined specimens for hot rolling had dimensions of 400×60×8 mm<sup>3</sup>. Each specimen had a lead to ensure an easy bite in rolling. Hot rolling tests were carried out on the 2-high Hille 100 experimental rolling mill with a roll diameter of 225 mm and a roll body length of 254 mm at the University of Wollongong. Before rolling, the 304 stainless steel workpieces were heat treated at 1100 °C for 1 h. The produced nanolubricant was sprayed onto the surface of the work rolls prior to each hot rolling test. The reduction was 30% and the rolling speed was 0.36 m/s. All the specimens were placed immediately in a cooling box with nitrogen gas and the top surfaces were stacked by a new blank specimen after rolling to prevent further oxidation and scale burst. The tests under each condition were done three times and then the mean values were obtained.

Table 1 Chemical compositions of stainless steel specimen (wt.%).

Steel	C	Cr	Ni	Mn	Si	P	Cu	V	N	Fe
304	0.047	18.09	8.45	1.13	0.51	0.027	0.046	0.12	0.027	Balance

Several small samples with a size of 15 (rolling direction) × 12 (width) × 5.6 (thickness) mm<sup>3</sup> were cut from the hot rolled specimen for observation and measurement.

The specimen surface and oxide scale cross-section morphologies were analysed by a VK-X100 laser scanning microscope and a JEOL JSM-7001F thermal field emission scanning electron microscope (SEM) with a large area energy-dispersive X-ray spectroscopy (EDS) detector.

### 3. Results

Fig. 1 shows the rolling forces and the surface roughness of the hot rolled specimen under different lubrication conditions. The average rolling force under dry, O/W, 0.5TiO<sub>2</sub>, 1TiO<sub>2</sub>, 1.5TiO<sub>2</sub>, 2TiO<sub>2</sub>, 4TiO<sub>2</sub> and 6TiO<sub>2</sub> conditions are 238.9 kN, 229.6 kN, 226.3 kN, 225.3 kN, 224.0 kN, 224.4 kN, 225.2 kN and 225.5 kN, respectively. The average surface roughness of the specimen under dry, O/W, 0.5TiO<sub>2</sub>, 1TiO<sub>2</sub>, 1.5TiO<sub>2</sub>, 2TiO<sub>2</sub>, 4TiO<sub>2</sub> and 6TiO<sub>2</sub> conditions are 1.06 μm, 0.96 μm, 0.93 μm, 0.86 μm, 0.84 μm, 0.86 μm, 0.88 μm and 0.89 μm, respectively. It is clear that the application of the nanolubricants can effectively reduce the rolling force and improve the strip surface quality, as compared with the conventional O/W emulsion. In addition, the rolling force and surface roughness decrease first, and then reach the minimum value at the nanoparticle mass fraction of 1.5%, and finally increase when the nanoparticle mass fraction in the O/W fluid is further increased.

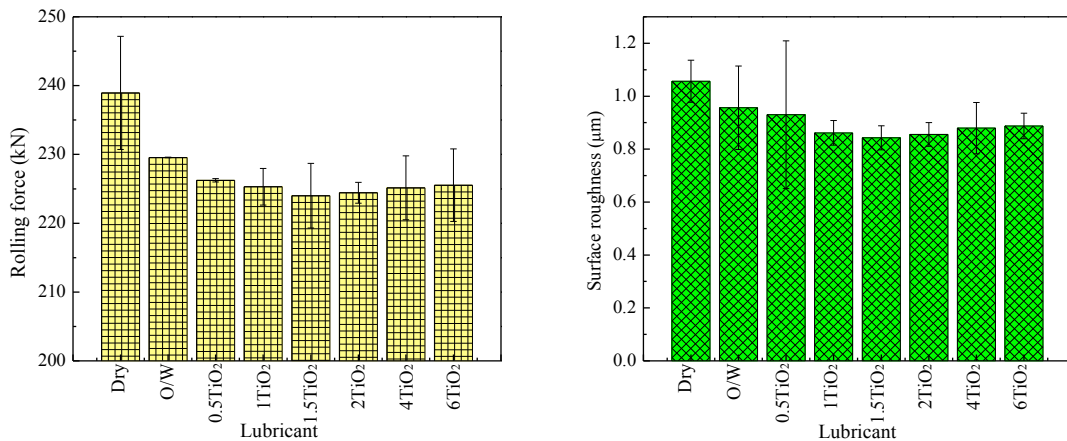


Fig. 1. Effect of lubrication conditions on (a) rolling force and (b) surface roughness.

Fig. 2 shows the cross-sectional morphologies of the hot rolled specimen under different lubrication conditions. It can be seen that the oxide scale formed on the hot rolled specimen under dry lubrication is thin and discontinuous. The oxide scale layer of the specimen under O/W lubrication becomes thicker. The oxide scale layer of the specimen becomes continuous and thicker once the nanolubricants were used. In addition, an increased nanoparticle mass fraction in the O/W fluid will cause the thicker oxide scale generated on the hot rolled specimen.

Fig. 3 shows that the surface morphologies of the hot rolled specimen under different lubrications. There are many long crack-like valleys on the surface after testing under dry lubrication, as shown in Fig. 3(a). The length of these valleys becomes shorter and their number decreases when the O/W lubricant is used, as compared with Fig. 3(a) and (b). The application of nanolubricants can effectively prevent these valleys to generate (see Fig. 3(c) and (d)).

The SEM images and EDS mapping element spectrums under dry, O/W, 1.5TiO<sub>2</sub> and 4TiO<sub>2</sub> lubrications are shown in Fig. 4. It can be seen that the oxide scales are broken and pressed after rolling under all lubrication conditions (marked with arrows). Flower-like oxide scale can be generated after O/W lubrication and nanolubrication. The nanolubricants can promote the generation of oxide scale, and an increase in the nanoparticle mass fraction of O/W fluid causes an increase in the area of the flower-like oxide scales (marked with circles in Fig. 4(b)-(d)). The EDS mapping element spectrums show that the nanoparticles can retain on the surfaces of the rolled specimen with a higher Ti element mass fraction when the nanolubricant with a higher TiO<sub>2</sub> nanoparticle mass fraction is used.

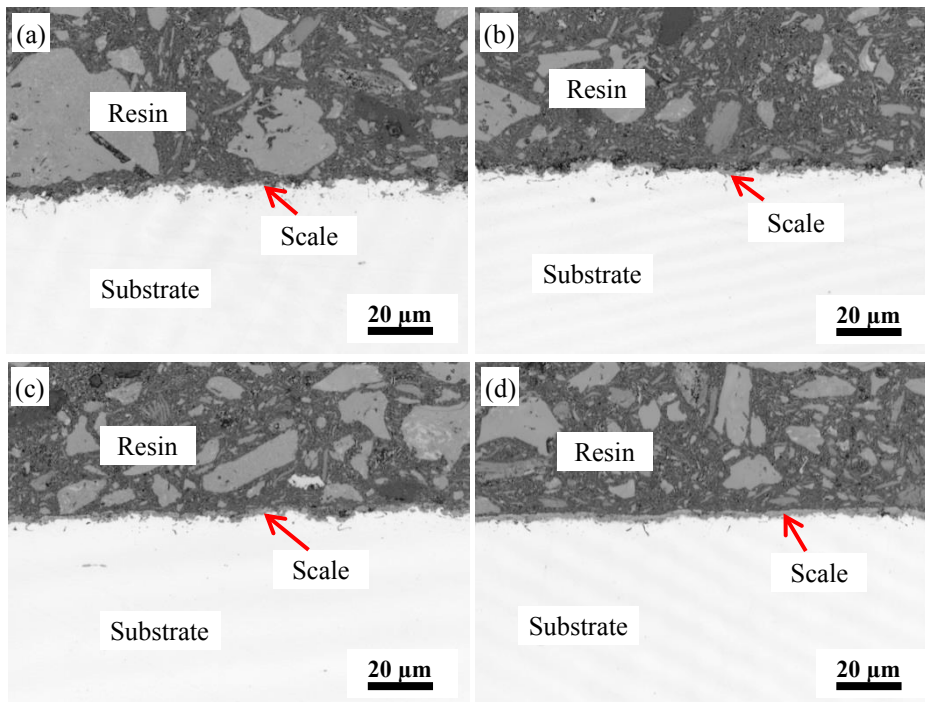


Fig. 2 Cross-sectional morphologies of the hot rolled specimen under different lubrication conditions: (a) dry, (b) O/W, (c) 0.5TiO<sub>2</sub> and (d) 4TiO<sub>2</sub>.

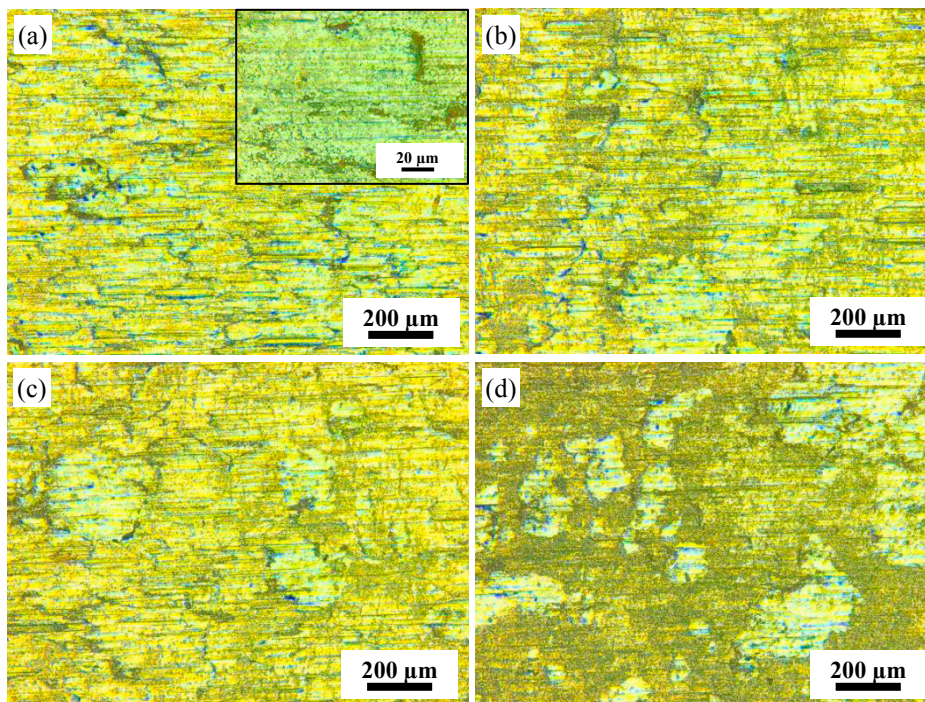


Fig. 3. Surface morphologies of the hot rolled specimen under different lubrications: (a) dry, (b) O/W, (c) 0.5TiO<sub>2</sub> and (d) 1.5TiO<sub>2</sub>.



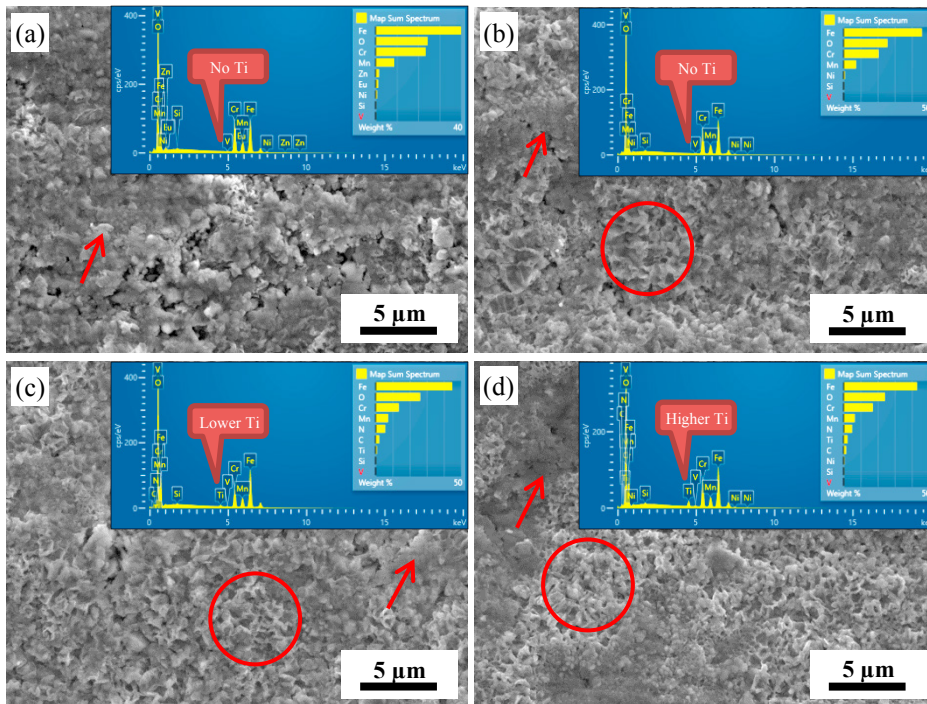


Fig. 4. SEM images and EDS mapping element spectrums under (a) dry, (b) O/W, (c) 1.5TiO<sub>2</sub> and (d) 4TiO<sub>2</sub> lubrications.

#### 4. Discussion

The rolling force decreases first and then increases with an increase of nanoparticle mass fraction in the O/W fluid (Fig. 1(a)). There is a positive relationship existed between the rolling force and coefficient of friction (COF) during rolling process. Therefore, an increase in the nanoparticle mass fraction can cause a decrease first and then an increase in the COF during the hot rolling process. The high temperature circumstance during hot rolling process has a significant influence on the lubrication mechanism, but the contacting time between the lubricant oil and hot workpiece is not enough for lubricant oil to reach its burning point, thus the oil/water mixture still keeps in liquid [4]. Azushima et al. [2] investigated the lubrication mechanism in hot rolling, using a new hot rolling machine, and revealed boundary lubrication or thin film lubrication. It means that the oil working with the form of liquid in hot rolling process can become a thin oil film in hot rolling process. The authors' previous works [5,6] reported the effect of nanoparticle mass fraction in the nanolubricant on the COF at 80 °C using ball-on-disc tests and the similar tendency of COF was obtained. The lubrication mechanism of the nanolubricant in the hot rolling process can be shown in Fig. 5 [7].

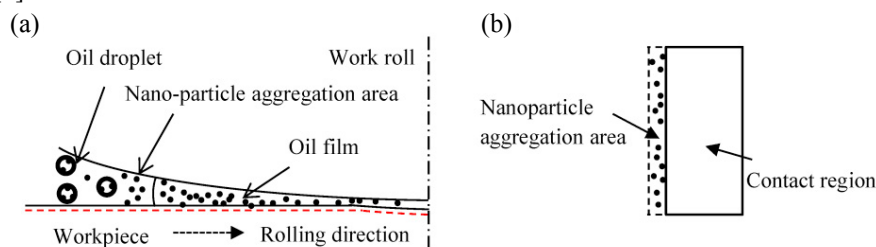


Fig. 5. Schematic diagram of (a) side and (b) top cross-section views of lubrication mechanism of the nanolubricant during hot rolling [7].

The addition of TiO<sub>2</sub> nanoparticles in the O/W lubricant can reduce the COF when the TiO<sub>2</sub> nanoparticle mass

fraction is below 1.5%. This is because the nanoparticles can enter the contact area and take an effect to reduce COF (see Fig. 4(c)). Once the mass fraction of the TiO<sub>2</sub> nanoparticles is over 1.5%, the extra nanoparticles cannot go through the contact area, and will become a nanoparticle aggregation area in the front of the entrance. This is because the oil film is very thin and the surface of workpiece is smooth, which cause difficulties for the extra nanoparticle to enter the generated oil film and the valleys on the surface of workpiece. The nanoparticles existed in the aggregation area can rub against each other, resulting in an increase of the COF. These nanoparticles can finally retain on the surface of the workpiece and more nanoparticles can be found on the surface of specimen when the nanolubricant with a higher TiO<sub>2</sub> nanoparticle mass fraction was used, as compared with Fig. 4(c) and (d).

The oxide scale layer of the specimen becomes thicker after the use of the conventional O/W emulsion, as compared with Fig. 2(a) and (b), but it is still thin. The thin scale can be broken and cracks can be generated along the width direction during hot rolling. The hot rolled specimen will be shortly oxidised in the air and reacted with the lubricant oil/water after rolling test, therefore, the crack-like valleys can be generated along the width direction on the surface of the specimen (see Fig. 3(a) and (b)). The application of the nanolubricant can improve the generation of a continuous and thicker oxide scale layer (see Fig. 2(c) and (d)), and prevent to generate the crack-like valleys (see Fig. 3(c) and (d)). The tendency of the area of the flower-like oxide scale (see Fig. 4(b)-(d)) also shows that the nanolubricant is beneficial to generation of oxide scale on the surface of 304 stainless steel. The continuous and thicker oxide scale layer on the surface of 304 stainless steel can cause reduction of rolling force further during the following hot rolling process [8]. The tendency of surface roughness is thought to be caused by the combined nanoparticle polishing and oxidation effects.

## 5. Conclusion

In this study, the effects of the O/W based nanolubricants containing TiO<sub>2</sub> nanoparticles in the hot rolling of 304 stainless steel were investigated. This study shows that the rolling force can be reduced and the surface roughness can be improved with the presence of TiO<sub>2</sub> nanoparticles in O/W based lubricant. An increased TiO<sub>2</sub> nanoparticle mass fraction can cause a decreased rolling force and surface roughness when the mass fraction of nanoparticle is below 1.5%. Above that, the rolling force and surface roughness will increase. The TiO<sub>2</sub> nanoparticles in O/W based lubricant can improve the generation of oxide scale on the 304 stainless steel surface.

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