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EFFECT OF SILICON CARBIDE NANOPARTICLES SIZE ON FRICTION PROPERTIES OF ELECTROLESS NICKEL COATINGS

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Abstract. The nickel coatings were produced by electroless plating process, without and with addition of 5–7 vol.% SiC nanoparticles of five different sizes (10, 45, 100, 150 and 700 nm in diameter). Coatings were tested in as-deposited and in heat treated (heating at 300°C for 6 h) condition, with two different surface textures preparation. This type of coatings was previously tested and showed improved abrasive wear resistance. Coefficients of friction testing of electroless nickel coatings in contact with bronze counter-body were determined in ambient air and unlubricated conditions. Static and kinetic coefficient of friction, as well as the difference between them, were analysed and discussed. The influence of size of embedded SiC particles in composite coatings, on tested friction properties of these coatings was also determined.

Keywords: electroless nickel coatings, composites, silicon carbide, nanoparticles, friction.

AIMS AND BACKGROUND

The latest trends in the development of tribology and tribotechnologies are related to the development of new ecological tribomaterials and coatings with high wear resistance, which do not contain toxic and carcinogenic substances[1,2]. Non-plated nickel coatings with nanosized particles of different materials have higher resistance to wear and corrosion, which makes them a serious competitor to chrome-plated coatings. It is well known that electrolyte chromium coatings are considered to be coatings with the best qualities, but their production is associated with hazardous waste for human health and the environment. European Union directives are for the gradual restriction of industrial chromium, and in some countries it is already banned[3].

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Electroless plating is one of the quite often used coating deposition process and it can be categorised as solution state coating deposition processes, in which mainly chemical energy is applied for deposition of the material[4]. Among other materials, nickel is the most commonly used in electroless plating (electroless nickel plating, i.e. electroless nickel coatings)[5]. The deposition of nickel coating with electroless plating process is applied in industry mainly in situations where corrosion protection and/or wear resistance are required[6]. It includes chemical process, food, oil and gas, automotive, aerospace, electronics and other industries[7].

Very often phosphorus is integrated into the electroless nickel coatings, and its content affects in a great manner the coating structure and its physical, mechanical and tribological properties. According to their phosphorus content, four groups can be distinguished: low phosphorus (1–3 wt.%), low-medium phosphorus (3–6 wt.%), medium phosphorus (6–9 wt.%) and high phosphorus (9–13 wt.%) alloys[8]. In addition, electroless nickel coatings can be heat treated, which diminished corrosion resistance but significantly improve wear resistance[7]. Thanks to these features electroless nickel has been used, e.g. to replace chrome coatings, which have negative environmental and health effects.

Furthermore, electroless nickel coatings can be deposited with incorporated micro- and nano-sized particles, producing a composite coating. Theoretically, almost any type of particle could be incorporated if it could withstand the conditions within an electroless nickel bath. So far, in some of our previous studies, we proved enhanced abrasive wear resistance of electroless nickel coatings with embedded nanoparticles of silicon carbide[7,9,10], diamond[4,11,12] and boron nitride[4,13].

A review of the literature in this field shows that there is a lack of systematic research of the effect of silicon carbide nanoparticles on friction properties of electroless nickel coatings, so the aim of the presented study was to investigate the influence of size of embedded SiC particles on static and kinetic coefficient of friction of these coatings.

EXPERIMENTAL

MATERIALS

All Ni coatings were produced by electroless plating process, with the phosphorus content of 9–11 vol.% (high phosphorus alloys). Composite coatings were obtained with addition of 5–7 vol.% SiC nanoparticles of different size, i.e. 10, 45, 100, 150 and 700 nm in diameter. Scanning electron microscopy (SEM) image of SiC nanoparticles with 150 nm in diameter is presented in Fig. 1. The substrate material for all coatings was a carbon steel Ст3кп (GOST 380), in disk shape of 100 mm diameter and 2.5 mm thickness, with following chemical composition: Fe-0.4C-0.2Si-0.55Mn-0.3Cr-0.3Ni-0.45P-0.045S (wt.%). Microhardness of the substrate was 135 $HV_{0.5}$.

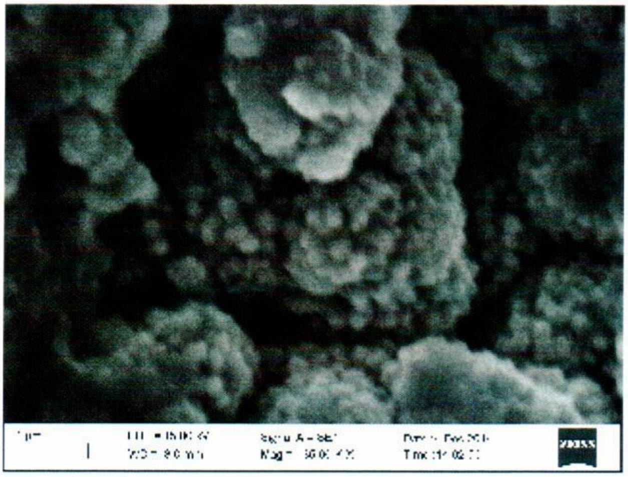

Fig. 1. SEM image of the SiC particles with 150 nm in diameter

Heat treatment (heating at 300°C for 6 h) was applied to half of the samples to improve its mechanical properties and adhesion in the substrate-coating interface. A total of 12 samples with different electroless Ni coatings were investigated. Two samples did not contain nanoparticles, and the remaining 10 coating samples contained SiC nanoparticles of different size. Designations and description of the tested samples are shown in Table 1. The thickness of the coatings was measured in 10 points by Pocket-LEPTOSKOP 2021 Fe, and the calculated average thickness values are also shown in Table 1. As it can be noticed, thicknesses were similar for all samples, with the average value of 25 μm.

Table 1. Designation and thickness of the tested samples

No	Designation	Description	Thickness (μm)
1	Ni	as-deposited coating without nanoparticles	25.5
2	Ni^{HT}	heat treated coating without nanoparticles	23.4
3	Ni-SiC10	as-deposited coating with 10 nm size SiC	25.3
4	Ni-SiC10^{HT}	heat treated coating with 10 nm size SiC	24.0
5	Ni-SiC45	as-deposited coating with 45 nm size SiC	23.6
6	Ni-SiC45^{HT}	heat treated coating with 45 nm size SiC	25.2
7	Ni-SiC100	as-deposited coating with 100 nm size SiC	25.8
8	Ni-SiC100^{HT}	heat treated coating with 100 nm size SiC	24.8
9	Ni-SiC150	as-deposited coating with 150 nm size SiC	25.6
10	Ni-SiC150^{HT}	heat treated coating with 150 nm size SiC	24.4
11	Ni-SiC700	as-deposited coating with 700 nm size SiC	27.6
12	Ni-SiC700^{HT}	heat treated coating with 700 nm size SiC	24.8

The microhardness ($HV_{0.5}$) measurements were performed using a Vickers microhardness tester under the load of 500 g and dwell time of 15 s. Five measurements were taken for each sample in order to obtain a representative value of the material microhardness. Results are shown in Fig. 2 and represent the average values. The repeatability of the results, in terms of standard deviations, was excellent, i.e. below 1.1 $HV_{0.5}$. Hardness of the referent coatings, i.e. coatings without SiC nanoparticles (Ni and Ni^{HT}) were in accordance with the literature data[4], and somewhat similar with the results of some previous studies[7,8]. Hardness of the coatings with SiC nanoparticles also were in the same range as in previous study[7] but were not the same. Differences from the previous studies could be due to the different phosphorous content, as well as due to the slightly different production process. However, the two things are the same. First thing is that all heat treated samples have higher microhardness than as-deposited (Fig. 2), which is due to the recrystallisation and grain growth during heat treatment[4]. The average increase of microhardness due to heat treatment was 173 $HV_{0.5}$, i.e. 29 %. Second thing is the influence of SiC nanoparticles size, i.e. small SiC nanoparticles (10 to 20 nm) have the most beneficial influence on microhardness, and with increase of SiC nanoparticles size microhardness start to decrease (Fig. 2). This is the general principle in as-deposited as well as in heat treated samples.

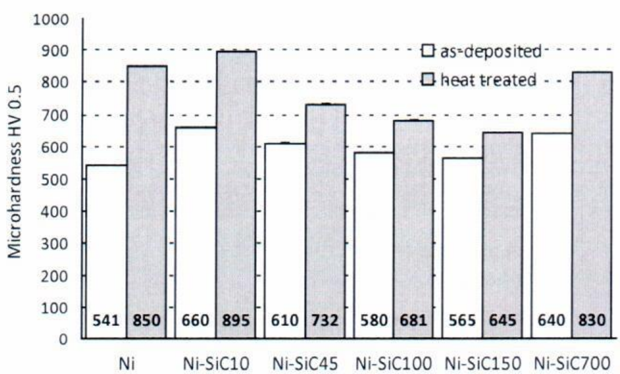

Fig. 2. Microhardness values and appropriate standard deviations of tested materials

Each of 12 samples from Table 1 was tested with two surface textures, i.e. initial texture (after coating deposition without machining) and working texture (after purposely roughening/smoothing of the samples). Purposely roughening/smoothing of the samples was done in order to simulate working, i.e. in-service conditions. It was done on Taber Abraser with abrading wheel Calibrase® CS-10 (Fig. 3a) and fixed load (2.45 N), sliding speed (approx. 0.24 m/s) and abrading time (900 s), giving the different visual appearance of the surfaces (Fig. 3b).

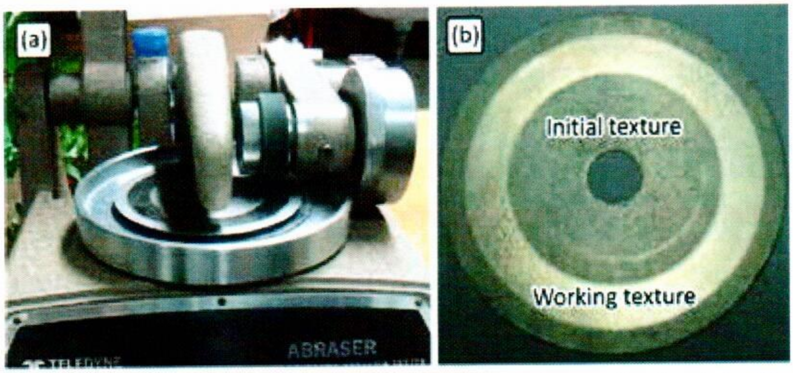

Fig. 3. Purposely roughening/smoothing of the samples: Taber abraser with abrading wheel = a and optical image of the working (lighter circular ring) and initial texture (rest of the disk) on the tested coated discs (coating Ni-SiC10) – b

The roughness of the samples was measured with a mechanical profilometer TESA Rugosurf 10G in two perpendicular directions and the mean value was calculated. These values were similar for all samples, so only the averaged values are presented: for initial texture it was $R_a = 0.418$ μm and for working texture it was $R_a = 0.396$ μm.

COEFFICIENTS OF FRICTION TESTING

The coefficient of friction testing was performed on the laboratory device, schematically presented in Fig. 4, which enables determination of the two values, i.e. static and kinetic coefficient of friction. Test sample (1) is stationary and fixed to the base. The counter-body (2) is in contact with the test sample (1), fixed in the holder (3) and connected through the non-elastic string with the dynamometer (6). The normal force (F_n) is set by means of the weights (4). Tangential force (T) is loaded to the counter-body near the contact surface through the very slow rotation (sliding speed of 10 μm/s) of the micrometric screw (5) and displayed on the dynamometer (6). Five measurements of each, static and kinetic friction force, were taken for every sample in order to obtain representative values.

Test parameters were as follows: normal load of 5.9 kg (57.9 N); dry contact condition, in ambient air at room temperature (≈ 25°C) and relative humidity of 40–45%. Counter-body was made of bronze (264.4 $HV_{0.05}$), and the geometrical contact area of counter-body with test sample was approximately 78.5 mm^2. Considering this contact area, the specific load was approximately 0.74 MPa. The resting time (for the possible stress relaxation at the junctions) was 60 s.

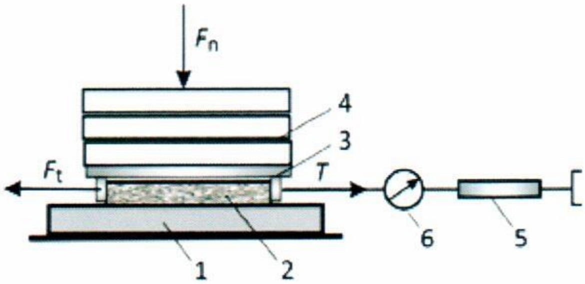

Fig. 4. Schematic diagram of the coefficients of friction testing

RESULTS AND DISCUSSION

The obtained values of the friction forces are presented in Fig. 5. It is obvious that the results are in line with the general theory, i.e. that the static coefficient of friction is greater than the kinetic one. The repeatability of the results, in terms of standard deviations, also was very good, i.e. below 3%. The influence of heat treatment was relatively small, i.e. applied heat treatment increased the microhardness of samples (Fig. 2), which induces lower real area of contact and decreased the adhesion component of friction, but in the same time it also increased the ploughing component of friction (counter-body material was relatively soft)[14]. Since the heat treated samples showed higher friction force (both, static and kinetic) than as-casted samples, it seems that the ploughing component increase had higher influence. Statistically, the increase in friction due to heat treatment was approximately 9%, and it was slightly lower for static friction (7.8%) than for kinetic friction (11.1%).

The obtained values of the surface roughness, presented through the arithmetic mean deviation of the assessed profile (R_a), were very similar for initial and working conditions (0.418 and 0.396 μm). Therefore, friction values were also similar for initial and working texture. Nevertheless, there are some differences in both, static and kinematic friction values. The static friction force of samples with working texture on average had lower values by 12% compared to samples with initial texture. This is in accordance with the accepted theory, which claims that contact surface roughness has direct influence on the static friction coefficient, and static coefficient of friction value increases if contact surfaces have bigger roughness parameters[15,16]. On the other hand, kinetic friction force of samples with working texture, in most cases had higher values compared to samples with initial texture (average increase was 10%). This could be due to the different distribution of the material in the surface layer, spacing or some other parameters which have decisive influence on kinematic friction[15–17]. The effect of surface texture on the tribological properties is discussed in details elsewhere[18].

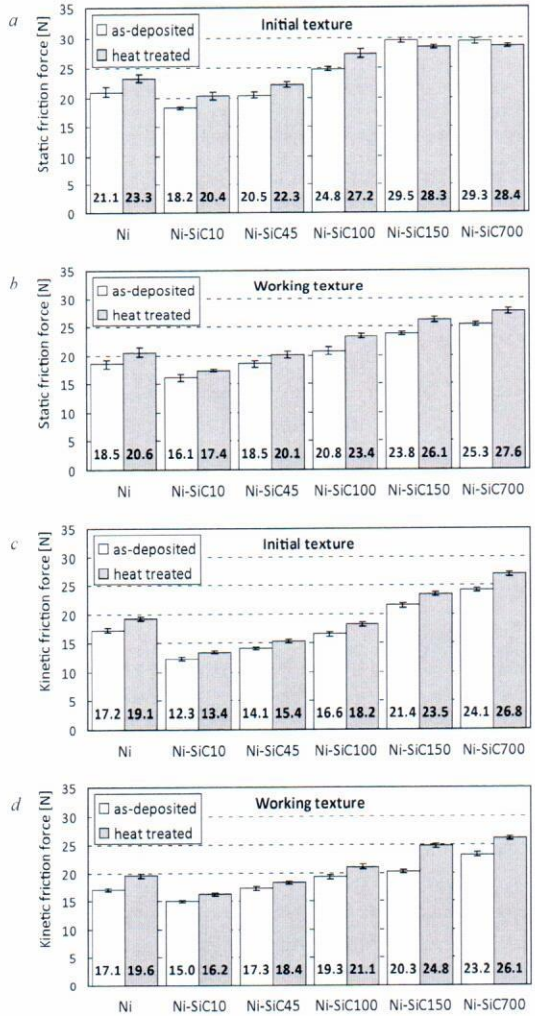

Fig. 5. Static and kinetic friction force values and appropriate standard deviations of as-deposited and heat treated samples (contact pairs): initial and working texture: diagram of the static friction force of the tested coatings without and with heat treatment for initial texture (before wear) – *a*; diagram of the static friction force of the tested coatings without and with heat treatment for working texture (after wear) – *b*; diagram of the kinetic friction force of the tested coatings without and with heat treatment for initial texture (before wear) – *c*, and diagram of the kinetic friction force of the tested coatings without and with heat treatment for working texture (after wear) – *d*

The influence of size of embedded SiC nanoparticles on static and kinetic coefficient of friction is presented in Fig. 6. Values of the coefficient of friction were in the range 0.28–0.51 for static coefficient of friction, and 0.21–0.46 for kinetic coefficient of friction. These values more or less correspond to the experimental values for metal-metal contact under dry sliding conditions[17]. All obtained coefficient of friction versus SiC nanoparticles size dependence graphs (Fig. 6) have similar appearances. Both coefficients of friction, for all samples, decreased with the addition of the smallest SiC nanoparticles (with size of 10 nm in diameter). After that, they start to increase as the SiC nanoparticles size increase. This increase was almost linear and very intensive up to SiC nanoparticles size of 150 nm, and after that the increase is also linear but less pronounced. This means that only small sizes of SiC nanoparticles have the beneficial influence on friction properties, which is in accordance with the regularity noticed for microhardness values (Fig. 2).

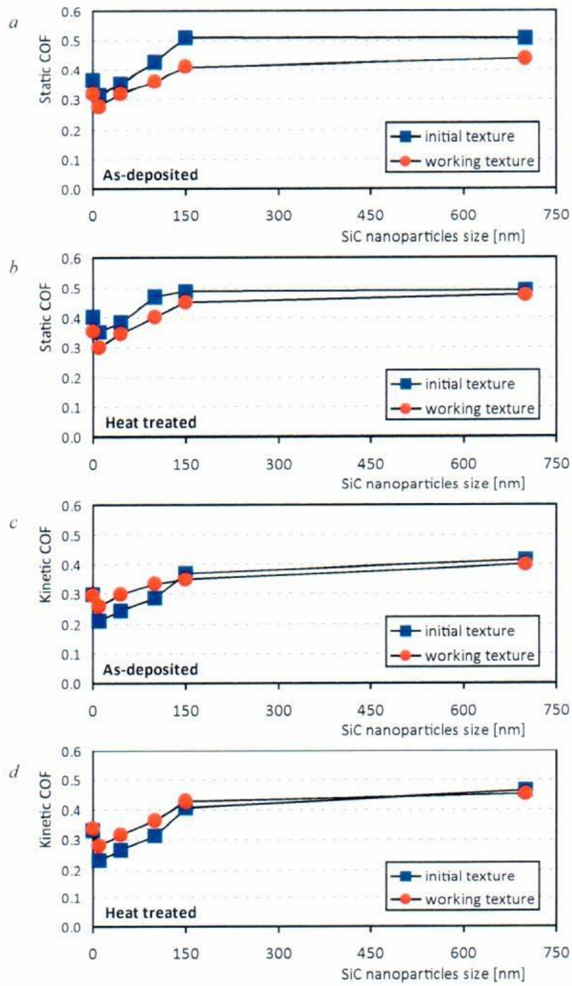

Fig. 6. Static and kinetic coefficient of friction (COF) values of different texture samples (contact pairs): as-deposited and heat treated: dependence of the static coefficient of friction on the size of SiC nanoparticles for coatings without heat treatment before and after wear – *a*; dependence of the static coefficient of friction on the size of SiC nanoparticles for coatings with heat treatment before and after wear – *b*; dependence of the kinetic coefficient of friction on the size of SiC nanoparticles for coatings without heat treatment before and after wear – *c*, and dependence of the kinetic coefficient of friction on the size of SiC nanoparticles for coatings with heat treatment before and after wear – *d*

The lowest coefficient of friction is noticed for samples with embedded SiC nanoparticles of smallest size (Ni-SiC10/Ni-SiC10^{HT}), and the highest coefficient of friction is noticed for samples with embedded SiC nanoparticles of largest size (Ni-SiC700/Ni-SiC700^{HT}). In comparison to the samples without nanoparticles (Ni/Ni^{HT}), the average decrease of coefficient of friction in the first case was 18%, and the average increase of coefficient of friction in the second case was 35%. The thing that must be emphasised when analysing the friction results is that the main reason for addition of SiC nanoparticles was improvement of coatings wear resistance. Although it did not increase the erosive wear resistance[19], it was shown that the abrasive wear resistance can be significantly increased[9,10].

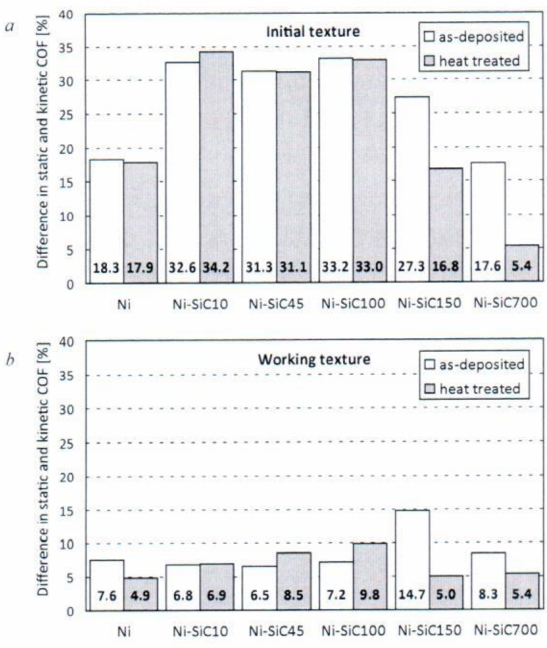

Fig. 7. Differences between static and kinetic coefficient of friction (COF) values of as-deposited and heat treated samples (contact pairs): initial and working texture: differences between static and kinetic coefficient of friction (COF) values of as-deposited and heat treated samples for initial texture (before wear) – *a*, and differences between static and kinetic coefficient of friction (COF) values of as-deposited and heat treated samples for working texture (after wear) – *b*

The calculated differences between static and kinetic coefficient of friction values are shown in Fig. 7. These differences did not differ too much between the samples and it seems that heat treatment have very small or no influence at all. In contrary to this, it is obvious that texture of the samples has high impact on differences between static and kinetic coefficient of friction. The average decrease of kinetic comparing to static coefficient of friction was 0.10 (24.9%) for samples with initial texture, while for samples with working texture this average decrease was

0.03 (7.6%). Static coefficient of friction is usually greater than kinetic coefficient of friction of about 20 to 30% (Ref. 20), and samples with initial texture are more or less in these limits. On the other hand, very small differences for samples with working texture show the beneficial influence of the purposely produced texture process, which in a way represents a running-in of the mating surfaces.

CONCLUSIONS

Applied heat treatment increased microhardness of all tested electroless nickel coatings by approximately 29%, which is correlated with the recrystallisation and grain growth during heat treatment. Heat treatment also induces higher values of both, static and kinetic, friction forces by approximately 9%. Higher hardness implies lower real area of contact and decrease the adhesion component of friction, but in the same time it also increased the ploughing component of friction (counter-body material was relatively soft). Heat treatment had very small or no influence at all on the calculated differences between static and kinetic coefficient of friction.

The applied running-in of the contact surfaces of electroless nickel coatings in which the surfaces were smoothed (initial vs. working texture) showed an effect of a double-nature on friction properties. The static friction of samples with working texture was lower by approximately 12% compared to samples with initial texture. On the other hand, kinetic friction of samples with working texture, in most cases had higher values compared to samples with initial texture (average increase was 10%). The process of running-in showed the biggest beneficial influence on differences between static and kinetic coefficient of friction. The average decrease of kinetic comparing to static coefficient of friction was 25% for samples with initial texture, and for samples with working texture was only 8%.

The influence of size of embedded SiC nanoparticles on friction properties was also of a double-nature. Both, static and kinetic coefficient of friction, for all samples, decreased with the addition of the smallest SiC nanoparticles (with size of 10 nm in diameter). After that, they start to increase as the SiC nanoparticles size increase. This increase was almost linear and very intensive up to SiC nanoparticles size of 150 nm, and after that the increase is also linear but less pronounced. This means that only small sizes of SiC nanoparticles have the beneficial influence on friction properties, and the lowest coefficient of friction is noticed for samples with embedded SiC nanoparticles of smallest size. In comparison to the samples without nanoparticles the average decrease of coefficients of friction for these samples was 18%. Addition of SiC nanoparticles and its size had very small or no influence at all on the calculated differences between static and kinetic coefficient of friction.

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