

**THE USE OF BOUNDARY LUBRICANTS FOR THE REDUCTION OF PARTICLE
JAMMING IN ABRASIVE PARTICLE SLURRIES FOR JET CUTTING**

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ABSTRACT

THE USE OF BOUNDARY LUBRICANTS FOR THE REDUCTION OF PARTICLE JAMMING IN ABRASIVE PARTICLE SLURRIES FOR JET CUTTING

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The development of an abrasive slurry jet (ASJ) is an important area of research in the abrasive water jet cutting industry. Direct pumping of abrasive slurries allows for more efficient use of the available energy of the high-pressure water, leading to faster cutting speeds, reduced cut kerf, and the potential to cut thicker materials. The development of ASJ technology requires the use of additives to create stable suspensions (against sedimentation) that are easily mixed, transported, stored, and pumped, and in particular that alleviate the problem of particle jamming in flow constrictions. This work investigates the effectiveness of surfactant boundary lubricants to increase lubricity and relieve jamming in model aqueous slurries of 50- μm diameter garnet abrasive particles. Lateral force microscopy (LFM) is used to characterize the lubricity of garnet surfaces pre-treated with cationic or neutral surfactants or functionalized with an octadecylsilane. All three treatments are found to produce significant increases in lubricity. The increases in particle surface lubricity are found to correlate with the results of macroscopic jamming tests in which garnet particle slurries are made to flow through the constriction provided by a syringe. Reductions of approximately 50% in jamming were achieved even under high particle loading conditions using the silane surface functionalization.

INTRODUCTION

The development and commercialization of abrasive water jets (AWJ) in the early 1980s represented a significant step forward for machining and cutting of materials [1]. One of the biggest advantages of the technology is that pieces cut using AWJ do not suffer from heat warping near the cut surface, as often occurs with laser cutting. This is particularly important for the cutting of composite materials, where the presence of a heat-degraded zone in the final product can lead to a drastic deterioration of the material properties and create possible failure points. In addition, AWJ technology offers the advantages of easily adjustable cut widths and no dulling of the cutting tool, making it a versatile and dynamic cutting tool. However, current AWJ technology suffers from several drawbacks. The performance of AWJ treatment depends strongly on the behavior of the liquid jet [1-3]. Modern abrasive water jets function by creating a low-velocity flow of ultra-high pressure (UHP) water. The water then passes through a gem orifice into a mixing chamber, which accelerates the water. Dry abrasive particles are introduced into the mixing chamber, entrained into the jet using the Venturi effect, and then accelerated out of the nozzle to strike the work piece. This process results in only approximately 10% momentum transfer to the particles [4], i.e., not all the kinetic energy of the particles is used to treat the work piece due to inefficient power transfer. In addition, the axial particle spacing in the jet is roughly an order of magnitude larger than the particles themselves [4]. Attempts have been made to solve these issues using increased abrasive particle flowrates into the mixing chamber; however, it has been found that this results in an inverse relationship with particle velocity [1, 4].

Additional experiments with AWJ in the early 1980s led to the conclusion that the inefficiencies of power transfer to abrasive particles and sparse particle spacing within abrasive jets could be solved by the development and implementation of abrasive slurry jets (ASJ) [5, 6]. In contrast to the method of entrainment of dry abrasive particles into a high-pressure jet that is currently used in AWJ technology, an ASJ employs direct pumping of a premixed abrasive

suspension, or “slurry” [5, 7]. This results in almost 100% efficient momentum transfer to the abrasive particles and controllable particle spacing by changing the concentration of abrasive particles in the slurry. The results of these improvements are increased cutting ability, increased cutting speed, and finer kerf width for a given horsepower jet. To this point, however, ASJ technology has not been commercialized due to issues in formulating a stable abrasive slurry so that particles would remain in suspended form (with respect to sedimentation) because the abrasive particles have a high specific gravity resulting in rapid settling. Various polymeric gelators have been found to address the problem of particle sedimentation, whereas the present work addresses the problem of particle jamming.

In this study, we explore the effect of lubricating abrasive garnet particle surfaces with adsorbed surfactants, viz. the cationic hexadecyltrimethylammonium bromide (HTAB) and the nonionic poly(ethylene oxide)(PEO) – poly(propylene oxide)(PPO) triblock copolymer Pluronic L62, and by surface functionalization with octadecyltrichlorosilane (OTS). In each case, the treatments will produce a lubricated surface [8]. We have sought to quantify the decreased friction using lateral force microscopy (LFM) and to qualitatively assess the effect of the surface treatments on garnet abrasive particle jamming as the slurries of treated abrasive particles are made to flow through a constriction.

MATERIALS AND METHODS

Materials

Flat-surfaced 0.5 cm square garnet pieces for LFM measurements were purchased from Allstarco.com. Garnet abrasives (220 mesh size, average diameter 50 μ m) were obtained through Alibaba.com, and a micrograph of the particles is shown in Fig. 1.

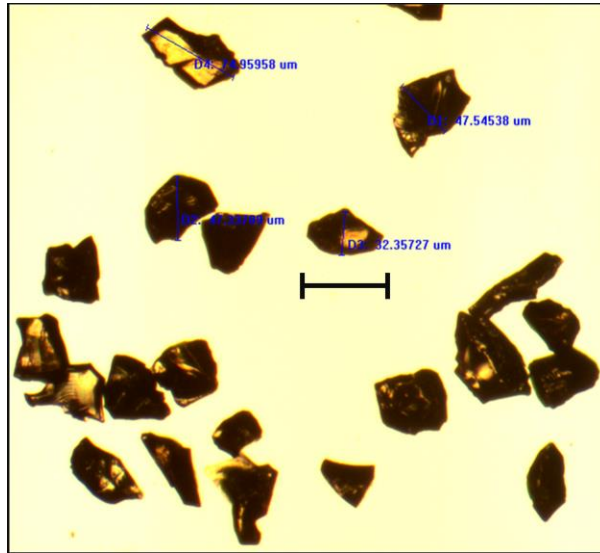


Figure 1: Micrograph of garnet abrasive particles

Hexadecyltrimethylammonium bromide (HTAB) and Pluronic L62 (PEO MW=1000) were obtained from Sigma Aldrich (St. Louis, MO) and BASF Corp. (Florham Park, NJ) respectively. Octadecyltrichlorosilane was purchased from Gelest Inc. (Morrisville, PA).

Sample preparation

Both the large garnet pieces and abrasive particles were first cleaned with isopropyl alcohol followed by air drying. The large garnet pieces used for the LFM measurements and abrasive particles used in the jamming tests were surface treated in the same way. The garnet was placed in the surfactant solution of interest for 15 min for adsorption of surfactant to the surface. Concentrations of 0.1, 0.3, 0.6, 0.7, 0.8, 1.2, 1.5, 2, 3, 4, and 5mM were used for the HTAB and 1, 2, 3, 5, 7, 12, 16, and 20mM for the Pluronic L62. Chemical modification of the garnet surface with OTS was carried out in a 1% v/v solution of isopropyl alcohol prepared in

advance and allowed 60 min for hydrolysis of the silane. The garnet was then placed in the solution for 90 min for complete reaction of the silane with the surface. During the process, pH was maintained at 4.5 using acetic acid. During the surface reaction, the silane covalently bonded with the garnet surface. The process occurs via a hydrolysis reaction, which forms silanol groups, followed by a condensation reaction with other silanol groups [9, 10] as shown schematically in Fig. 2. Following treatment, the garnet was removed from the solution, cleaned with isopropyl alcohol, air dried, and the samples used for LFM measurements or the jamming tests.

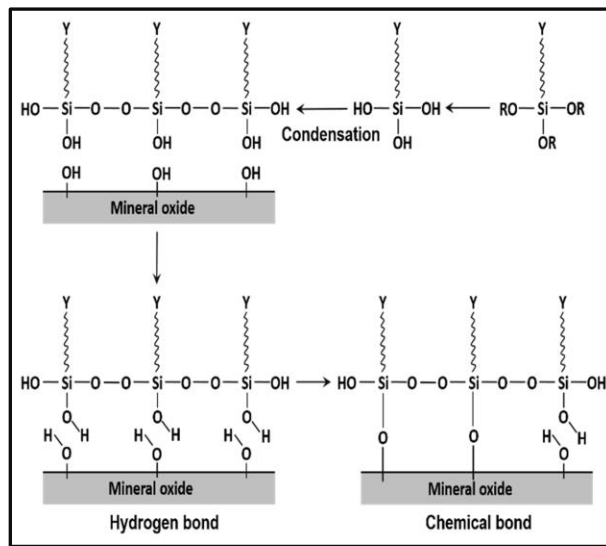


Figure 2: Surface modification using organofunctional silanes

Lateral force atomic force microscopy (LFM)

Changes in local friction against a garnet surface caused by the various surface treatments used were assessed using lateral force microscopy [11]. A sharp tip of silicon nitride affixed to a flexible cantilever is pressed against the garnet surface and moved back and forth across the surface with a scan velocity of 0.2 nm/s in the lateral force (LF) mode within a scanning range of 1 μm . Both the normal force pressing the tip against the garnet surface and the resistance to lateral motion can be measured once the normal and lateral spring constants, k_N and k_L of the

cantilever are known, and the instrument is calibrated, i.e., its sensitivity is determined. As the cantilever moves over the surface, torsion bending of the cantilever takes place. Information is obtained by measuring the cantilever deflection, which is determined as the horizontal signal of a quad-cell photodetector. The lateral (friction) force is measured for various normal forces. Both are indicated by a signal given in nano-amperes (nA) and calculated using Eq. (1). For calculating the load applied, the set point provided is used as the signal while for calculating the friction force, the half amplitude of the lateral signal produced by the oscilloscope in response to the friction, is used as the signal.

$$\text{Force (N)} = \frac{(k_N \text{ or } k_L) \frac{\text{N}}{\text{m}} \cdot \text{Signal (nA)}}{\text{Sensitivity} \frac{\text{nA}}{\text{m}}}. \quad (1)$$

The experimental setup for the LFM is shown schematically in Fig. 3, which shows the instrument, a Veeco Topometrix Explorer (Fremont, CA) employing a cantilever from Nanoscience Instruments Inc., (Phoenix, AZ) (Spring Constant 1 N/m). An oscilloscope was used to monitor the lateral signal of the photodiode, i.e., a square wave, for different setpoints provided. All measurements were performed at ambient temperature and pressure. In order to obtain accurate friction force, both forward and reverse scans were necessary, which produced a square wave, the half amplitude of which is proportional to the friction force. For measurements in liquid (water), the sample was placed in a liquid cell.

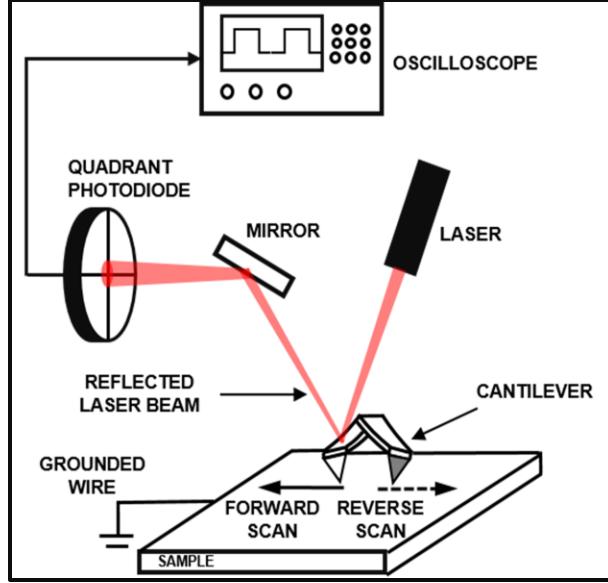


Figure 3: Experimental setup for lateral force microscopy (LFM)

Calibration of the tip, in order to determine the normal and lateral (friction) forces, was performed as described elsewhere [11]. The out-of-contact cantilever resonance frequency, f_c (in Hz), in the vertical direction is required to determine the cantilever thickness, t , and was obtained prior to friction measurements using Eq. (2)

$$t = 0.000723L^2 f_c, \quad (2)$$

where L is the cantilever length. The cantilever thickness was then used to obtain the normal and lateral spring constants, k_N and k_L , in conjunction with the known cantilever dimensions and moduli): tensile modulus, E , and shear modulus, G , according to Eq. (3) and Eq. (4) respectively [10].

$$k_N = \frac{Et^3W}{4L^3}, \quad (3)$$

$$k_L = \frac{Gt^3W}{3Lr^3}, \quad (4)$$

where W , L and t are the cantilever width, length, and thickness, respectively and r is the tip height. Cantilever dimensions such as width, length and tip height were provided by the manufacturer.

The sensitivity (nA/nm) of the photodiode was calculated as the slope of the force-distance curve of the cantilever whose tip is pressed against a silicon wafer, with the force signal given in terms of a current (nA). Prior to friction measurement of each sample, the corresponding adhesion force was computed from the AFM force-distance curve so as to calculate the total normal force (F_n) which is the combination of adhesion force and the load applied. Thus, friction force was plotted against the normal force, and the friction coefficient was determined as the slope of linear fit to this plot. For all the LFM measurements, the set point range of 0-1.8 nA ($F_n \sim 0$ -2.4 μ N) was used as beyond this range for most of the samples, the lever crashed into the sample leading to unusable results.

Macroscopic jamming measurements

A simple macroscopic test was devised to test the efficacy of the various particle surface treatments. Slurries consisting of 1.41, 2.00, 2.67 and 3.42 g of particles mixed with 8 mL of water or surfactant solution were put into a 1 cm i.d. syringe, to which a needle of 100 μ m i.d. was attached, as pictured in Fig. 4. The concentrations used produced solids loadings of 15, 20, 25, and 30% w/w. Without any pressure applied to the slurries, capillary forces were sufficient to retain the mixture in the syringe. Upon application of a downward pressure by means of the syringe plunger, however, slurry could be made to flow through the syringe needle. Application of a weight of 400 g to the top of the plunger produced (accounting for the weight of the plunger itself) a pressure of 5 kPa to the slurry. While this amount of pressure was unable to produce flow from the slurries of the untreated particles, flow was often, but not always, produced in the

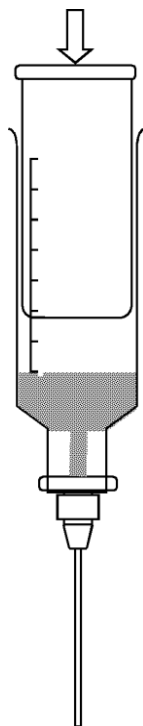


Figure 4: Schematic of macroscopic particle jamming tests.

case of the treated particles, suggesting that in these cases, the jamming of the particles was relieved by the lubricity produced by the particle surface treatments. When outflow was observed, it was always a slurry, i.e., a mixture of the particles and the liquid. For the surfactant trials, concentrations of 0.005M HTAB and 0.012M Pluronic L62 were used, approximately their critical micelle concentrations, at which it is assumed that close-packed monolayers are formed. 20 trials were performed for each system, and the fraction of the trials in which jamming was relieved was recorded for each case.

RESULTS AND DISCUSSION

LFM Friction tests

The friction force of the LFM tip against the bare garnet surface and the surface treated with adsorbed films of the two surfactants, HTAB and Pluronic L62, as a function of the surfactant concentration, as well as the surface functionalized with a film of the OTS, were determined. The lateral resisting force, F_f , was measured for each case as a function of the normal force, F_n , as shown in Fig. 5(a) for the case HTAB adsorbates. A reasonably linear dependence of F_f on the normal force F_n was obtained for all cases, and it was clear that both the absolute magnitude of the lateral resisting force and its slope were reduced by the presence of the surfactant, acting as a boundary lubricant. The slope of the lateral vs normal force is taken as the measure of friction, μ , and is shown in Fig. 5(b). Figures 6(a) and (b) show the similar results obtained for the Pluronic L62 surfactant. In both cases, it is seen that the friction was reduced sharply, by more than a factor of two, and exhibited a shallow minimum in a particular concentration range. For HTAB, the minimum occurred near $C \approx 1$ mM, while for Pluronic L62 at $C \approx 7$ mM, both values in rough correspondence to their respective critical micelle concentrations (*CMC*) of 0.95 mM for HTAB [12] and 9.6 mM for Pluronic L62 [12]. Garnet, which is a silica variation, has a negative surface charge in aqueous media at pH values above approximately 2.5 [13], so that a cationic surfactant such as HTAB adsorbs in a head-down configuration. With reference to Pettersson et al. [14], below the *CMC*, individual adsorption

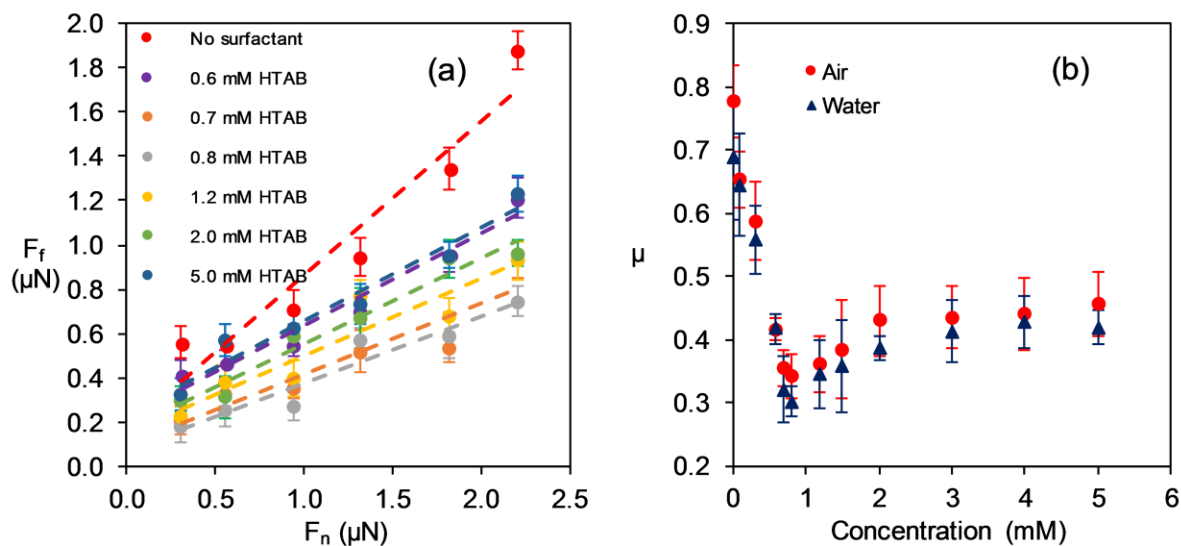


Figure 5: (a) Friction force, F_f , vs normal force, F_n , for different concentrations of HTAB; (b) Friction coefficients, μ , calculated as the slopes of curves in (a).

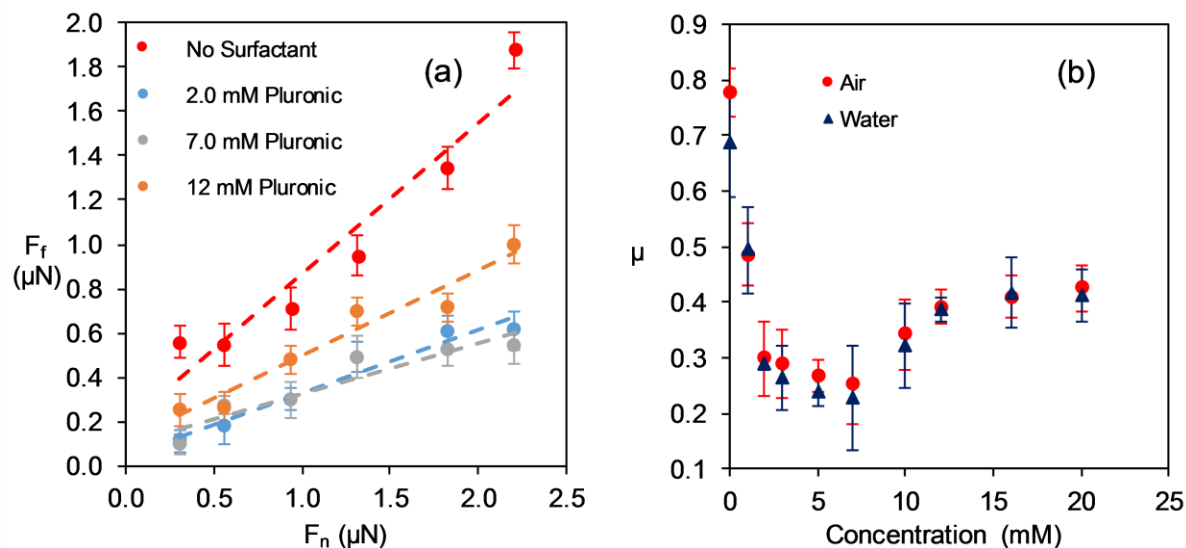


Figure 6: (a) Friction force, F_f , vs normal force, F_n , for different concentrations of Pluronic L62; (b) Friction coefficients, μ , calculated as the slopes of curves in (a).

from the solution to the surface takes place until near the *CMC*, where the first monolayer is complete. Beyond the *CMC*, the second layer starts to form with tail-down adsorption, leading to a slight increase in friction factor as the cantilever tip must move through a thicker layer. Beyond

a concentration of ≈ 2 mM, the bilayer is complete, so that no further adsorption was expected, and no further change in friction coefficient with concentration was observed.

As noted, a similar trend in the friction factor was observed for the nonionic surfactant Pluronic L62, as shown in Fig. 6. This surfactant is a triblock copolymer with terminal hydrophilic poly(ethylene oxide) (PEO) groups and a central block of hydrophobic poly(propylene oxide) (PPO) groups which provide the adsorption anchor. As the concentration of the Pluronic was increased, the friction factor decreased sharply until about 0.002M, a value much lower than its *CMC* (0.0096M) in water, after which the friction factor gradually decreased before ultimately increasing slightly. Pluronic L62 is believed to begin forming hydrophobic domains, micelle-like surface aggregates at a concentration of 0.002M [15-18]. As the concentration approaches the *CMC*, a second layer starts to form, leading to an increase in the friction factor, until about 0.012M. For concentrations above 0.012M, the friction coefficient is essentially constant as the second layer is completed. In summary, both surfactants were able to achieve significant friction coefficient reductions, which were maximized at concentrations near their respective *CMCs*.

An alternate strategy for achieving boundary lubrication of the garnet surfaces was to functionalize them with an organofunctional silane, specifically octadecyltrichlorosilane (OTS). Figure (7) shows the results obtained from friction measurements with LFM in water. The linear

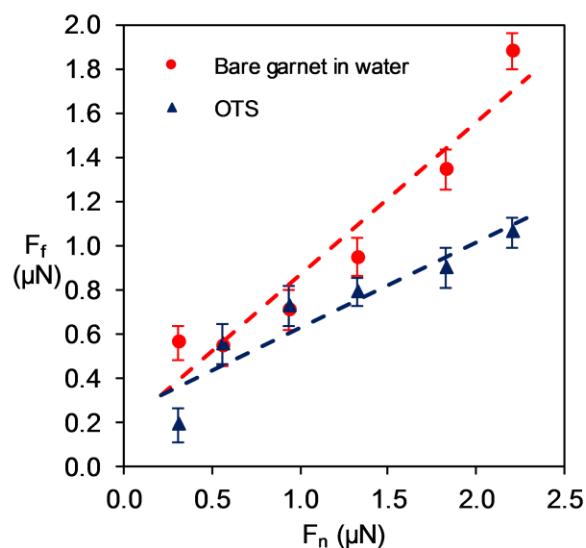


Figure 7: Comparison of straight line fit of friction force, F_f , as a function of normal force, F_n , for bare garnet and surface modified garnet.

fit to the friction force versus applied load plot showed that the slope, i.e., friction coefficient, was reduced from 0.68 to 0.38 with silane modification compared to the unmodified, bare garnet in water. Significant reduction in friction suggests that surface modification with OTS provided a good lubrication to the garnet surface by forming strong covalent bonds to the surface while exposing the hydrocarbon moieties to the surface, as shown in Fig. 2.

Jamming tests

Since the LFM tests suggested that large reductions between garnet particles were achievable using both adsorbed surfactants and silane surface modification, macroscopic jamming tests with the un-modified and the surface modified garnet abrasive particles were performed as described above. Tests were performed with variable loadings. Figure (8) shows results obtained with HTAB and Pluronic L62 at concentrations corresponding to those showing the highest drag reduction in the LFM tests. It can be seen that at low abrasive loadings, jamming

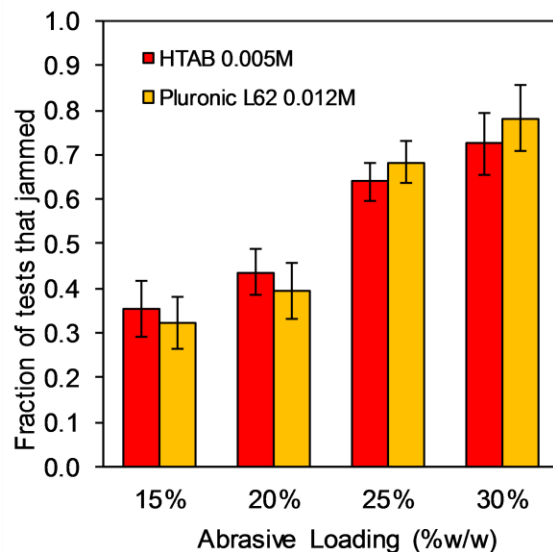


Figure 8: Comparison of jamming test results with different surfactants for variable abrasive loadings.

was significantly reduced, but when abrasive loading was as high as 25% to 30%, the fraction of the trials which jammed increased considerably, indicating that surfactant treatments may not be effective at these higher loadings. Figure (9) shows the comparison between jamming performance for bare and OTS-treated garnet particles in water.

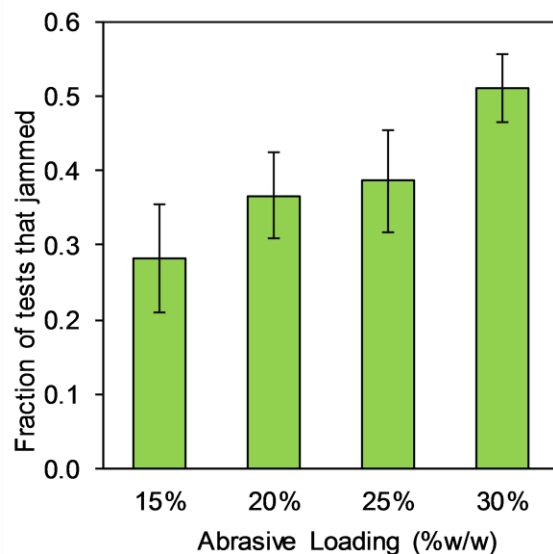


Figure 9: Jamming test results obtained for OTS functionalized garnet particles.

It is seen that even for the highest particle loadings, a significant reduction in jamming (about 50%) is achievable. In contrast, the untreated particles showed 100% jamming at all loadings.

CONCLUSIONS

It has been demonstrated that LFM friction measurements provide a useful method for quantifying the extent of lubrication of abrasive garnet particles via adsorption of surfactants like HTAB and Pluronic L62 to the garnet surface or by functionalizing the surface with OTS. Reduction in friction by about half was achieved with all three surface treatments. Macroscopic jamming tests with treated and untreated garnet abrasive particles indicated significant relief of particle jamming in all cases, but the surfactant treatments were less effective than the silane functionalized surfaces at high particle loadings.

In summary, this study has shown the potential effectiveness of abrasive particle surface boundary lubrication for the mitigation of particle jamming during water jet cutting. In particular, modification with octadecyltrichlorosilane has shown that jamming can be relieved even under conditions of high particle loading.

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REFERENCES

1. Wang, J. Review: Abrasive waterjet machining of engineering materials, *Scitech Book News*, Portland. **2003**, 24(4).
2. Hashish, M. A model for abrasive-waterjet (AWJ) machining. *J. of Eng. Mat. And Tech.-Transactions of ASME*. **1989**, 111(2), 154-162.
3. Momber, A.; Kovacevic, R. Principles of Abrasive Waterjet Machining. Springer. **1998**, 77-81.
4. Summers, D.; Bushnell, D. Preliminary experimentation of the design of the water jet drilling device. 3rd International Symposium on Jet Cutting Technology, Chicago, IL, **1976**, 21-28.
5. Hollinger, R.; Perry, W.; Swanson, R. Precision cutting with a low pressure, coherent abrasive suspension jet. The 5th American Waterjet Conference, Toronto, Canada. **1989**, 245-252.
6. Howells, G.W. Polymer blasting with super-water from 1974 to 1989: a review. *International Journal of Waterjet Technology*. **1990**, 1, 1-16.
7. Fair, J.C. Development of High-Pressure Abrasive-Jet Drilling. *J. Pet. Technol.* **1981**, 8, 1379-1388.
8. Berg, J. C. An Introduction to Interfaces & Colloids: The Bridge to Nanoscience. Hackensack, N.J.: World Scientific. **2010**, 196-197.
9. Louis, C.; Ullien, D.; Mescher, M.; Sudholter, E. Organic surface modification of silicon nanowire-based sensor devices. *Sensors*. **2013**, 14, 245-271.
10. Jal, P.; Patel, S.; Mishra, B. Chemical modification of silica surface by immobilization of functional groups for extractive concentration of metal ions. *Talanta*. **2004**, 62, 1005-1028.

11. Knorr Jr., D. B.; Gray, T. O.; Overney, R. M. Intrinsic friction analysis – Novel nanoscopic access to molecular mobility in constrained organic systems. *Ultramicroscopy*. **2009**, *109*, 991-1000.
12. Mukerjee, P.; Mysels, K. Critical micelle concentrations of aqueous surfactant systems. *Nat. Stand. Ref. Data System., Nat. Bur. Stand. (U.S.)*. **1971**, pp. 57.
13. Behrens, S.; Grier, D. The charge of glass and silica surfaces. *J. Chem. Phys.* **2001**, *115*, 6716-6721.
14. Pettersson, A.; Rosenholm, J. Adsorption of alkyldimethylamine and alkyldimethylphosphine oxides at curved aqueous solution/silica interfaces, studied using Microcalorimetry. *Langmuir*. **2002**, *18*, 8436-8446.
15. Sarkar, B.; Venugopal, V.; Tsianou, M.; Alexandridis, P. Adsorption of Pluronic block copolymers on silica nanoparticles. *Colloids and Surfaces A: Physicochem. Eng. Aspects*. **2013**, *422*, 155-164.
16. Ligoure, C. Surface micelles formation by adsorption of block copolymers. *Macromolecules*. **1991**, *24*, 2968-2972.
17. Tiberg, F.; Jonsson, B.; Lindman, B. Ellipsometry studies of the self-assembly of nonionic surfactants at the silica-water interface – kinetic aspects. *Langmuir*. **1994**, *10*, 3714-3722.
18. Eskilsson, K.; Tiberg, F. Interfacial behavior of triblock copolymers at hydrophilic surfaces. *Macromolecules*. **1998**, *31*, 5075-5083.