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Tribology of Soap Powders used in Dry Wire Drawing

by

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Declaration of Originality

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Abstract

Understanding friction and lubrication behaviour of powdered soaps used in dry wire drawing is crucial for reduced die wear, enhanced wire quality and improved energy efficiency. However, the complex tribological behaviour of powdered soaps proves to be challenging in providing a thorough understanding of the tribological interactions at the wire-die contact. In this work, lab tests were used to obtain accurate quantitative data on powdered soap friction, and to study the nature of soap lubrication.

To gain insights into the frictional behaviour of typical powdered soaps under conditions relevant to the wire die-contact in wire drawing, a systematic test campaign with selected soap chemistries was conducted using a high-pressure ball-on-disc tribometer. The impact of high sliding, speed and contact pressure on friction was explored using materials similar to die-wire materials.

Friction tests showed that powdered soaps provide a very good level of lubrication with friction coefficients (COF) being as low as 0.04 at high sliding-speeds and pressures. An increase in pressure and sliding-speed leads to a decrease in soap COF. At low speeds and low SRRs soap COFs are very variable suggesting potential effects of shear-rate. Soap formulation, particularly the amount of fatty acid content, was shown to be significant for friction performance. Measurements on a real wire drawing set-up showed that observed friction trends are in line with trends of drawing stress.

Soap lubricating films were studied using optical techniques which gave evidence of thick, residual soap layers on the rubbing track of the specimens. A high-speed optical ball-on-disc machine was used to directly observe in-contact soap film formations. Powdered soaps exhibit a very dynamic film behaviour with occasional formation of a constriction near the outlet, resembling the characteristic 'horse-shoe' shape of EHL fluids. It is postulated that the in-contact temperature rise has crucial effects on film formation.

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Nomenclature

Symbol	Unit	Description
A	m ²	Cross-sectional area of wire
A _c	m ²	Contact area
A ₀	m ²	Initial cross-sectional area of wire (entering die)
A ₁	m ²	Cross-sectional area of drawn wire (exiting die)
a	m	Contact half width or contact radius
B	-	Heat partition factor
b	m ² /s	Thermal diffusivity of wire
C	J/kg·K	Specific heat capacity
COF	-	Coefficient of friction
d ₀	m	Initial wire diameter (entering die)
d ₁	m	Drawn wire diameter (exiting die)
E	Pa	Young's modulus
E*	Pa	Reduced young's modulus, see equation (2.3)
F	N	Friction force
F _b	N	Backpull force
F _d	N	Drawing force
F _s	N	Separating force in Split-Die
$\bar{G}$	-	Dimensionless material parameter
h	m	Film thickness
h ₀	m	Minimum film thickness
h _{ciso}	m	Central film thickness, e.g. equation (2.11)
k _d	W/m·K	Die thermal conductivity
k	W/m·K	Thermal conductivity
L	m	Surface profile length
L _b	m	Bearing length (die)
L _c	m	Die contact length
L _d	m	Length of deformation zone (die)
L _t	m	Total length of wire-die contact
l	m	Wire length

m	-	Coefficient of pseudoplasticity of soap
P	Pa	Normal pressure / die pressure
P_m	Pa	Mean die pressure
$P_{\max}$	Pa	Maximum Hertzian pressure in contact
P_{mean}	Pa	Mean Hertzian pressure in contact
p	Pa	Pressure in lubricant film
Pe	-	Peclet number
$\dot{Q}$	W/m ²	Heat flux
$\dot{q}$	W	Heat rate
R	m	Radius of contact area
R^*	m	Reduced radius of relative curvature, see equation (2.9)
R_x^*	m	Reduced radius along rolling direction
R_y^*	m	Reduced radius transverse to rolling direction
R_0	m	Initial wire radius (entering die)
R_1	m	Drawn wire radius (exiting die)
r	-	Area reduction ratio
r^*	m	Polar radius in the contact plane, see equation (2.2)
R_a	m	Average roughness asperity height over centre line
R_q	m	Root mean square roughness
SRR	%	Slide-roll-ratio; ratio of sliding speed to entrainment speed
T	°C	Set temperature in ETM tests
T_b	°C	Bulk temperature
T_c	°C	(Total) Contact temperature, see equation (8.3)
T_{eq}	°C	Equilibrated temperature in wire
T_s	°C	Soap temperature
ΔT_f	°C	Flash temperature rise
$\Delta T_{\max}$	°C	Maximum surface temperature rise
ΔT_{mean}	°C	Mean contact temperature rise
T_0	°C	Wire temperature at inlet
$\bar{U}$	-	Dimensionless speed parameter
u	m/s	Velocity

U_a	m/s	Velocity of body A (see chapter 8.1)
U_b	m/s	Velocity of body B (see chapter 8.1)
U_{ball}	m/s	Ball speed
U_{disc}	m/s	Disc speed
U_e	m/s	Entrainment speed / rolling speed
U_s	m/s	Sliding speed
U_0	m/s	Wire speed at die inlet
U_1	m/s	Wire speed at die outlet
ν	-	Poisson's ratio
W	N	Contact load
W'	N	Normal point force
$\bar{W}$	-	Dimensionless load parameter
W_d	J	Drawing work
W_{pl}	J	Plastic deformation work
w	m	Contact deformation
w_d	N/m ³	Drawing work per unit volume
w_f	N/m ³	Friction work per unit volume
w_r	N/m ³	Redundant work per unit volume
w_u	N/m ³	Uniform work per unit volume
z	m	Height of (rough) surface above centre line
α	°	Die semi-angle
β	-	Ratio between maximum die heating and measured die heating
γ	-	Coefficient of piezoviscosity
$\dot{\gamma}$	1/s	Shear rate
Δ	-	Shape of deformation
δ	-	Coefficient of thermoviscosity
η	Pa·s	Dynamic viscosity
η_0	Pa·s	Base viscosity
Λ	-	Lambda ratio (minimum film thickness/asperity height)
λ	-	Elongation ratio
ρ	kg/m ³	Density
σ_a	Pa	Average flow stress in wire

σ_b	Pa	Backpull stress
σ_d	Pa	Drawing stress
σ_i	Pa	Average flow stress in wire at die inlet
σ_m	Pa	Mean normal stress at the centreline of the wire
σ_o	Pa	Average flow stress in wire at die outlet
τ	Pa	Shear stress
ϕ	-	Redundant work factor
χ	m ² /s	Thermal diffusivity

Acronyms

Abbreviation	Description
CCD	Charge Coupled Device
DSC-TGA	Differential scanning calorimetry and thermogravimetric analysis
EHD	Elastohydrodynamic
EHL	Elastohydrodynamic lubrication
ETM	Extreme-traction machine
FA	Fatty acid (content)
FTIR	Fourier-Transform Infrared
IR	Infrared
MTM	Mini-traction machine
PTFE	Polytetrafluoroethylene
R&D	Research and Development
SEM-EDX	Scanning electron microscopy with energy dispersive X-ray spectroscopy
SLIM	Spacer layer imaging method
UWT	Universal wire drawing test machine
WC	Tungsten carbide

1 Introduction

The background to this research is introduced in this chapter. The aims and objectives of this project are outlined, and the structure of this thesis is described.

1.1 Context of Thesis

Steel wires play an important role in our modern world. The areas of applications of steel wires are extensive and some examples are shown in Figure 1-1. Every year, 76 million tonnes of steel wire rods are manufactured globally (1). A commonly used metal forming process to produce steel wires is wire drawing. In its simplest form, wire drawing can be described as the process of pulling wire through a conically shaped die during which the wire diameter is reduced. Two main processes of wire drawing are employed, wet wire drawing, where emulsions are largely used as lubricants, and dry wire drawing with powdered soaps as lubricants.



Figure 1-1: Engineering components where steel wires can be found: a) champagne bottle wire muselet (2), b) steel strands for cable stayed bridges (3), c) anchorage rope wires in oil platforms (4).

In recent years, the demand for high productivity in wire drawing manufacture, higher quality of the produced wires and reduction in energy usage has increased significantly. These demands are necessitating major improvements in the dry wire drawing process which can only be achieved through improved understanding of the mechanisms involved. Better understanding of the tribology of the wire-die contact forms a major part of this. During drawing, large frictional forces are acting on the wire where wire and die are in contact. These result in shear stresses that have a major impact on wire surface quality, die life, and the energy consumed during the process. The latter

entails a very significant cost and environmental aspect as wire drawing is a highly energy intensive process. The electricity usage in wire drawing is reported to be 1,150 kWh on average per ton of steel wire produced¹; rough estimations under the assumption that steel wire is made entirely by wire drawing indicate that the yearly global energy consumption of steel wire drawing is equivalent to around 30% of the total electricity consumption of the United Kingdom (5). Concurrently, factors such as urbanisation and growth in infrastructure development (6), are increasing the demand for wires and calling for improved manufacturing productivity and quality of the wires. Hence, optimising the wire production process entails major economic and environmental benefits.

To overcome these challenges to achieve potential energy savings and wire product improvements, understanding the tribological interactions at the wire-die interface is crucial. However, there is remarkably little research into the tribology of wire drawing, in particular the dry wire drawing with powdered soaps. Hence, friction and lubrication in wire drawing are not well understood. This can primarily be ascribed to the fact that wire drawing involves very complex tribological conditions, such as high contact pressures in the GPa range (7,8) needed to plastically deform the wire, very high sliding speeds of up to 25 m/s (9), and lubricants with complex tribological behaviour such as dry powdered soaps.

The complexity of the tribological conditions in dry wire-drawing lead to many questions such as the effect of speeds, pressures and shear-rate on friction, as well as friction on temperature rise. The latter might also play an important role in the ability of soaps to form a low shear stress lubricating film, but the underlying lubrication mechanisms and the nature of those soap films are not clear yet. Moreover, past literature in this field lacks the capabilities to provide accurate quantitative friction data of powdered soaps which is needed as input parameters in numerical simulations of dry wire drawing. The work presented here aims to give new insights into the friction and lubrication behaviour of powdered soaps in dry wire drawing and to build a groundwork for further research in this field.

¹ Internal report Bekaert

1.2 Aims and Objectives

The overall goal of this thesis was to gain understanding of friction and lubrication with soap powders under conditions pertinent to dry wire drawing. Given the complexity of the wire drawing process and lubricants, this study focussed on the severe conditions in wire drawing such as high pressures and high sliding speeds. The first objective of this thesis was to develop a controlled lab-test to study friction with powdered soaps under these harsh conditions. To achieve this, a ball-on-disc tribometer was employed which was capable of direct friction measurements at high pressures and relatively high sliding speeds. This method was also used to identify key parameters that affect friction.

With reliable friction experiments conducted, the next aim of this research was to understand the lubrication behaviour of powdered soaps. In a first step, this involved analysis of deposited soap layers on specimens using optical and chemical techniques. For deeper understanding of the lubrication behaviour, an optical ball-on-disc machine was used to directly observe the contact. The impact of temperature on soap lubrication was also studied. Moreover, the friction trends were compared to observations of drawing stress during tests conducted on a real wire drawing machine. All of this was used to provide new insights into the nature of dry wire drawing soap films.

1.3 Structure of Thesis

After the introduction, chapter 2 gives an overview of the literature pertinent to the tribology of dry wire drawing. A brief summary of the basics of friction and lubrication mechanisms is provided. The metal forming process ‘wire drawing’ and terms relevant to this process are described. Lastly, a summary of existing literature on friction and lubrication in dry wire drawing with powdered soaps is given and critically reviewed.

Chapter 3 depicts the research methodology and the experimental methods used to study tribological interactions with dry powdered soaps. This chapter entails all techniques used to measure friction and assess lubrication, as well as methods used to characterise the specimens and soaps.

Chapter 4 focuses on friction measurements with typical dry powdered soaps. The

effect of different process parameters, as well as material properties, on friction is studied. The results are discussed in the context of existing literature.

In chapter 5, the residual soap layers present on test specimens after the friction experiments are studied. Analyses of such films conducted with a series of optical and chemical techniques are described. The deposited soap films are also assessed with regard to their durability and persistency.

In Chapter 6, an optical machine is employed to get direct access into the contact. The entrainment of soap particles and build-up of soap films at high speeds is directly observed. Some high-speed friction approximations are shown and correlations between friction trends and film observations are discussed.

Chapter 7 presents experiments conducted on a real wire drawing machine. Surfaces of drawn wires are also examined chemically. The findings of previously described lab bench tests are compared to the results from these wire drawing experiments.

In chapter 8, potential effects of temperature on the formation of soap lubricating films are investigated. Flash temperature rises in the contact are calculated for the test conditions employed in the present study. Temperature-dependent phase transitions of soaps are considered and discussed.

Chapter 9 provides a general discussion to unify the findings of this research. A strong focus is on the complex nature of the soap films and the findings from friction measurements, film observations and temperature effects are used to postulate on possible mechanisms of powdered soap film formation. This chapter also discusses the limitations of this work.

Finally, chapter 10 summarises the main findings of this thesis. Based on these findings and limitations, some potential avenues for further work are suggested.

2 Literature Review

This chapter describes the existing literature relevant to this work. First, the relevant tribological context is described. Then, the process of wire drawing and pertinent parameters are explained. Finally, previous tribological studies in the context of wire drawing are summarised.

2.1 Tribology

Tribology is the “science and technology of interacting surfaces in relative motion and of related subjects and practices” (10,11). This term covers the study of friction, lubrication and wear and it is a key discipline in any application with moving parts in contact. To understand the underlying tribological interactions in the wire-die contact, the principles of contact mechanics, friction and lubrication need to be explained.

2.1.1 Contact Mechanics

Two bodies in contact deform depending on the force that they exert on each other. At small loads and/or large contact areas, contact pressures are relatively low, the deformation is elastic, and the two bodies return to their initial state once the load is removed. When the load is high, or contact area small, the contacting surfaces might deform plastically. Depending on the geometry of the contacting bodies, the contacts can be described as conforming and non-conforming or counter formal (12). In conforming contacts, the geometry of the contacting surfaces is such that they conform to each other so that the area of contact is large, for example, a flat on flat or journal bearing. Here, pressures are usually low and only elastic deformations take place. When the surfaces of the bodies are non-conforming, the contacting area is small, and the pressure is very high. In this case, a significant elastic or even plastic deformation takes place.

The pressure distribution and contact area in a counter formal contact of two elastic bodies can be described using the Hertz Theory (13). The assumptions made in this theory are as follows:

- The surfaces are smooth and non-conforming.

- There is no friction between the surfaces.
- The strains are small (elastic deformation).
- The considered contact is a half-space (at least one parabolic body) and the contact length is very small compared to the thickness of the bodies.

The deformation w at a given position (x,y) for an elastic half-space subjected to a normal point force W' is given by the Boussinesq solution (14):

$$w(x) = \frac{1}{\pi E^*} \frac{W'}{r^*} \quad (2.1)$$

$$r^* = \sqrt{(x - x')^2 + (y - y')^2} \quad (2.2)$$

where r^* is the polar radius in the contact plane. E^* is the reduced modulus which combines the mechanical properties Young's modulus E and Poisson's ratio ν of the two bodies as follows (13):

$$\frac{1}{E^*} = \frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \quad (2.3)$$

The displacement field can then be determined by integrating over the contact area:

$$w(x, y) = \frac{1}{\pi E^*} \iint \frac{P(x', y') dx' dy'}{\sqrt{(x - x')^2 + (y - y')^2}} \quad (2.4)$$

For a circular contact the pressure distribution is given by (12):

$$P(x, y) = P_{max} \left(1 - \frac{r^{*2}}{a^2} \right)^{0.5} \quad (2.5)$$

where P_{max} is known as the maximum contact pressure, or simply „Hertz stress“, and a is the contact half width or contact radius. The Hertzian maximum and mean pressure for a circular contact, and contact half width can then be determined with an applied

total force W :

$$P_{max} = \left(\frac{1}{\pi}\right) \left(\frac{6WE^{*2}}{R^{*2}}\right)^{\frac{1}{3}} = \frac{3}{2} \frac{W}{\pi a^2} \quad (2.6)$$

$$P_{mean} = \frac{W}{\pi a^2} = \frac{2}{3} P_{max} \quad (2.7)$$

$$a = \left(\frac{3}{4} \frac{WR^*}{E^*}\right)^{\frac{1}{3}} \quad (2.8)$$

where R^* is the radius of relative curvature which can be found by the sum of the curvatures of the two surfaces (R_1 and R_2 being the radii of curvature of the two bodies in contact):

$$\frac{1}{R^*} = \frac{1}{R_1^*} + \frac{1}{R_2^*} \quad (2.9)$$

2.1.2 Friction

Friction is the resisting force opposing the motion when one body moves over another (15,16). First investigations into friction were introduced by Leonardo da Vinci in 1492 (10,17), followed by Amontons in 1699 (18), and Coloumb in 1785 (19). In a dry contact, there are two central causes for friction, namely adhesion and deformation (16). In engineering applications, surfaces are never entirely smooth, due to roughness asperities. Adhesive friction is usually much larger and occurs as a result of bonds at a molecular level between the contacting asperities of the surfaces (20); adhesive friction force refers to the force required to shear these junctions (16,20). Friction due to deformation relates to the force necessary to plough the asperities of the harder surface through the softer countersurface (16).

Lubricants are used to reduce friction in the contact. In lubricated contacts where a fluid completely separates the two surfaces, adhesive friction is insignificant (16) and the frictional force resisting motion occurs as a result of shearing of the thin lubricant

film. In mixed lubrication, where the fluid film is thin and solid-to-solid asperity contacts occur, both adhesion and fluid shearing contribute to total friction.

According to the basic laws of friction, the friction force F is proportional to the nominal normal load W . The ratio between the friction F and the nominal load W is referred to as the friction coefficient COF (15,16):

$$COF = \frac{F}{W} \quad (2.10)$$

The coefficient of friction defines the magnitude of the frictional force and depends on the contacting materials (21).

2.1.3 Lubrication

Lubricants are used to form a separating lubricant film between the two contacting bodies in order to reduce shear stresses and therefore, friction and wear of the materials (22). The prevailing lubrication regime depends on the thickness of the lubricant film between two surfaces in relative motion in relation to the height of the roughness asperities on the surfaces. In general, three different lubrication states can be distinguished in fluid-lubricated contacts: boundary, mixed and hydrodynamic lubrication. A subset of hydrodynamic lubrication is elastohydrodynamic (EHD) lubrication that occurs in non-conformal contacts where pressure is high. The transformation process of the lubrication mechanisms can be visually characterised by the Stribeck curve as seen in Figure 2-1, where the friction coefficient varies with load W , viscosity η and velocity u (20). Usually, the lambda ratio Λ , which is the ratio of the minimal film thickness h_0 to some measure of asperity height (commonly the arithmetic average Ra) is used to describe these lubrication regimes (20).

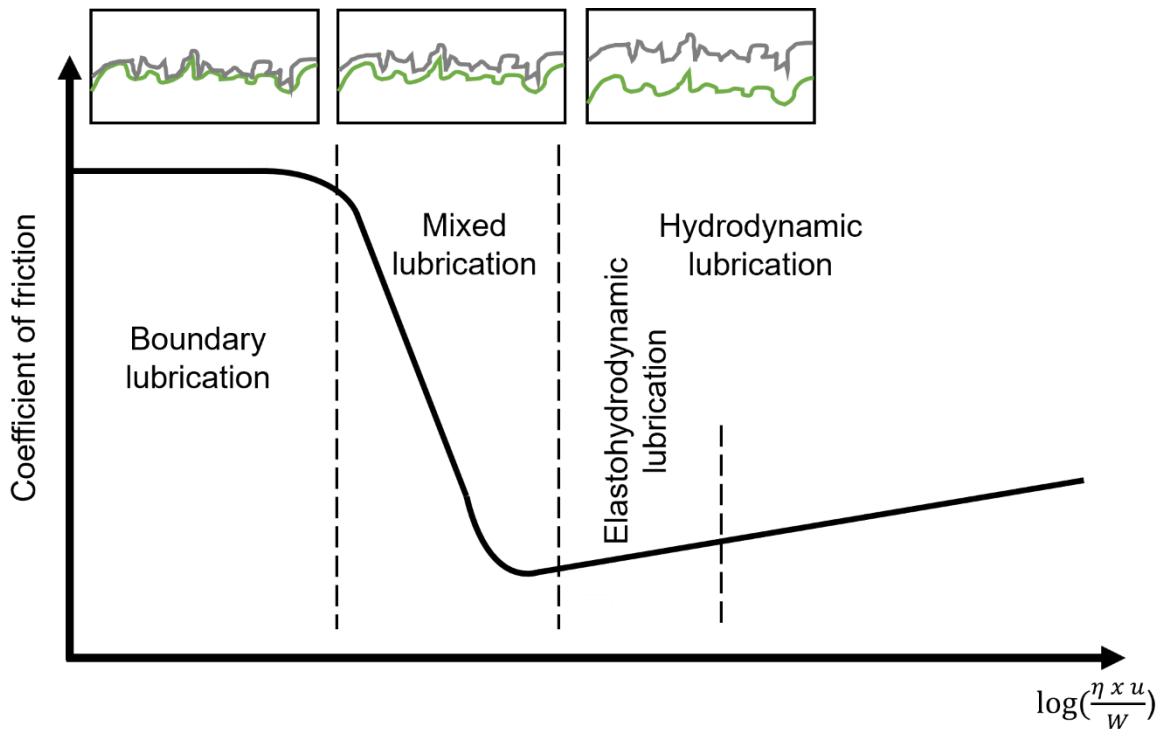


Figure 2-1: Stribeck curve and lubrication states

Hydrodynamic Lubrication

When a fluid lubricant separates the two surfaces in relative motion entirely, the hydrodynamic lubrication regime is effective (21). This regime occurs at high speeds (15,16), or high lubricant viscosity. High relative velocities and the lubricants viscosity lead to a pressure rise in the film which supports the normal load and prevents the opposing surfaces from contacting (15). This regime employs a thick lubricant layer where the film thickness, h , is much higher than the asperity heights, Ra , (20), with a lambda ratio greater than about 3 to 5 being indicative of full film separation. Many refer to this mechanism as the ideal form of lubrication since it separates the surfaces completely from each other (16).

The pressure distribution within a hydrodynamic fluid film can be characterised by the Reynolds' equation (15). This is derived from the Navier-Stokes equations for fluid flow assuming laminar flow, thin film and that viscosity causes prevailing forces (10,16). Solving the Reynolds equation, which requires adequate boundary conditions, allows for quantifying the lubrication conditions. However, the standard Reynolds' equation is limited by assuming a constant lubricant viscosity and a Newtonian behaviour of the

fluid (10,16). It also neglects inertia forces and the compressibility of the lubricant (10).

Elastohydrodynamic Lubrication

Elastohydrodynamic lubrication (EHL) is a subset of hydrodynamic lubrication and occurs in counterformal contacts where the local pressures in the fluid film are large, much larger than those generated under standard (nominally rigid) hydrodynamic lubrication (20). Under such conditions, both the dependence of the viscosity on the pressure and the variation of the film thickness through elastic deformation of contacting surfaces need to be considered (15), hence the name elastohydrodynamic lubrication. In this case, at least one of the surfaces undergoes significant elastic deformation and the contacting area grows (15,20). In addition, the lubricant viscosity increases with increasing pressure (15). Both these effects promote the formation of a lubricant film despite the very high contacting pressures (1 to 3 GPa for typical hard steels used in tribological components such as bearings and gears).

In cases where the contacting bodies are very elastically compliant (low elastic modulus), lower pressures are encountered despite the non-conformity of original surface geometry; Such pressures do not significantly increase the lubricant's viscosity and hence, only the elastic deformation of the contacting surfaces need to be taken into account (15). This lubrication regime is often termed 'soft EHL' or more precisely iso-viscous elastic. This is in contrast to piezo-viscous elastic behaviour of the standard EHL regime where surfaces have a relatively high elastic modulus (15).

The minimum and central film thickness of EHL films is usually predicted using various regression equations. For example, equations by Hamrock and Dowson (23) are widely used. Here, the film thickness is expressed through the dimensionless parameters of speed ($\bar{U}$), load ($\bar{W}$) and material ($\bar{G}$). Different equations exist for point, line and elliptical contacts. For central film thickness in general elliptical contact, Hamrock and Dowson provide the following expression (23):

$$h_{ciso} = 2.69\bar{U}^{0.67}\bar{G}^{0.53}\bar{W}^{-0.067}\left(1 - 0.61e^{-0.75k\left(\frac{R_y^*}{R_x^*}\right)^{0.64}}\right) \quad (2.11)$$

where R_x^* and R_y^* are the reduced radii along and transverse to rolling direction, respectively.

Boundary and Mixed Lubrication

When the contact conditions in fluid lubricated contacts do not allow for full film separation due to low velocities or low oil viscosity, the load is supported by a combination of fluid film and asperity contacts. When the oil film is negligible and practically all of the load is carried by the asperities (λ ratio < 1) boundary lubrication regime prevails (10,21). Under boundary lubrication, lubricant additives chemically react with the surfaces to form a very thin solid-like film on the surfaces which acts to reduce friction and wear (15). Boundary films are usually tens of nanometres in thickness and the friction is higher than under other lubrication conditions (20).

The mixed lubrication mechanism applies when the load is shared between asperities and fluid film, with λ ratio of between 1 and 3. It is an intermediate regime between the boundary and the hydrodynamic lubrication where friction and film parameters are affected by both fluid properties and the solid-solid contact regions (24).

2.2 Wire Drawing

Wire Drawing is the process of reducing the cross-sectional area of a wire, rod or bar by pulling it through a conical die with a smaller diameter. When no specific differentiation is needed, wire, rod and bar are referred to as “wires” in general as the analyses of those workpieces are analogous (25). The wire drawing process is schematically shown in Figure 2-2 where the wire is drawn from left to right.

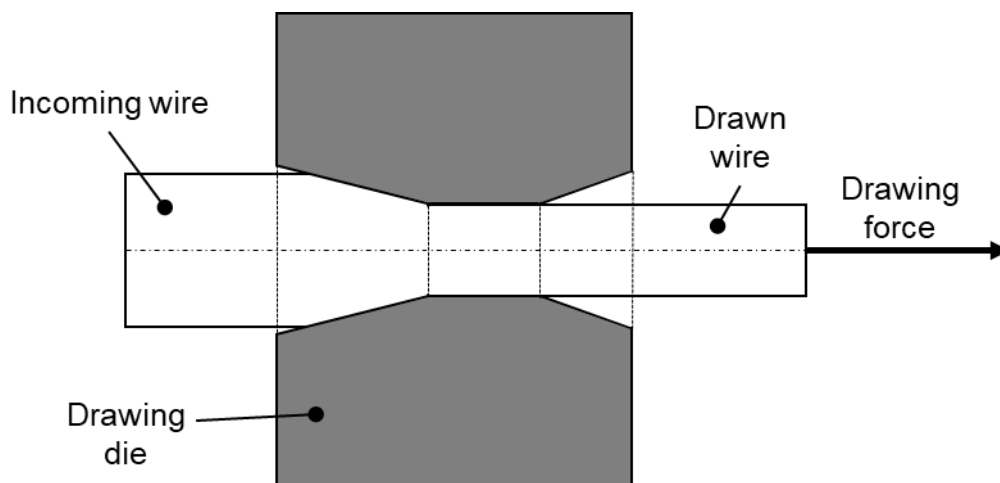


Figure 2-2: Illustrative scheme of wire drawing process adapted from (24) with permission of Cambridge University Press through PLSclear.

A pulling force is necessary to draw the wire through the die. This is provided by a rotating capstan that is located behind the drawing die and on which the wire will be wound (24,25). To produce thin wires at high speeds, multiple-die machines are most efficient (25). The spooled wire on the capstan will then be fed to the next drawing die with an even smaller diameter and is pulled by another capstan. By placing several drawing dies and capstans consecutively, a continuous reduction of the wire diameter can be achieved down to very thin wires of 0.1 mm in diameter (24). Because of the volume constancy, the cross-sectional reduction of the wire leads to an increase in wire length (24,25). With growing wire length, the drawing speed increases and hence, different angular velocities for the successive capstans are required (25). This cold-forming process causes strain-hardening of the wire which reduces ductility (24). To avoid breakage of the wire, it needs to undergo a soft annealing operation before further drawing which helps re-establishing the material's ductility (24).

An example of a multi-pass drawing system is illustrated in Figure 2-3 where the wire is drawn from left to right. For the first drawing pass, the wire enters the soap box from the first capstan. The drawing force is applied through the drawing capstan which draws the wire through the drawing die. After the final (n^{th}) drawing pass, the wire is recoiled again on another capstan. The drawing parameters are described in more detail below.

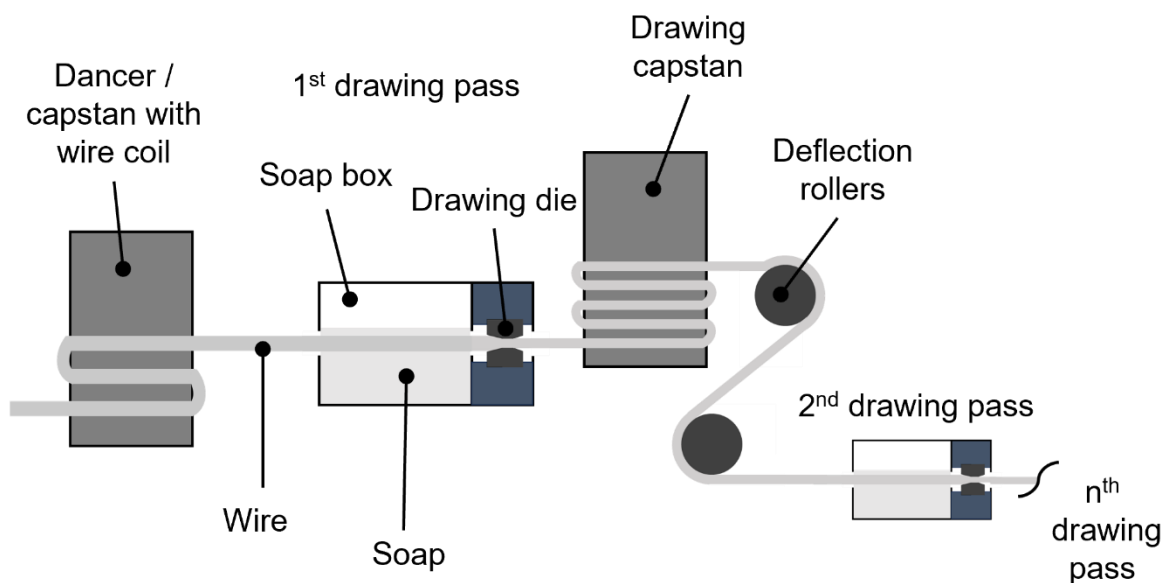


Figure 2-3: Exemplary image of a multi-pass drawing system with the wire being drawn from left to right.

2.2.1 Geometrical and Mechanical Parameters in Wire Drawing

An overview of the analytical parameters of the wire drawing process is illustrated in Figure 2-4, where the wire is drawn from left to right. The following sections describe each of the shown parameters.

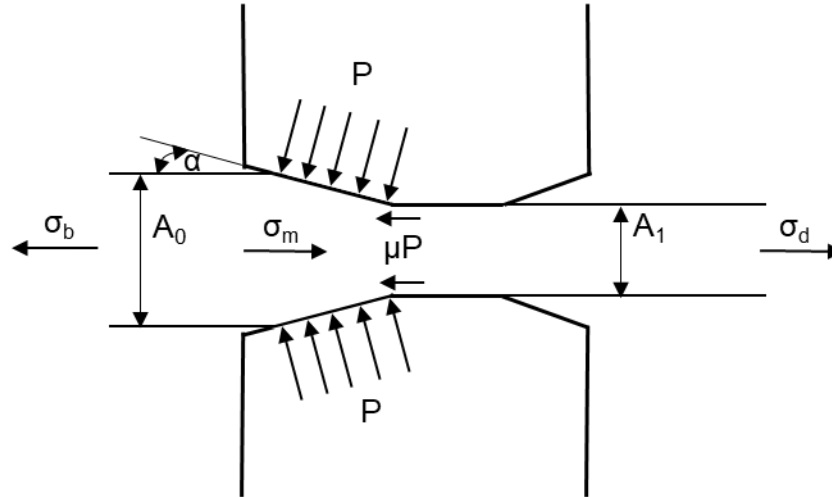


Figure 2-4: Schematic illustration of drawing parameters in the drawing pass adapted from (25) with permission from Elsevier.

Cross-sectional Area Reduction

During drawing, the wire undergoes a reduction of its initial cross-sectional area A_0 and exits the die with a cross-sectional area A_1 . This reduction r can be defined as follows (25):

$$r = \frac{A_0 - A_1}{A_0} = 1 - \frac{A_1}{A_0} \quad (2.12)$$

Die Angle and Deformation Zone

Plastic deformation occurs only when the wire passes through the converging area of the die (24). This drawing channel is characterised by the die's semi-angle α which is the angle between the inner conical die wall and the drawing axis (25). The deformation zone starts with the first contact of wire and die and ends at the end of the cone where the parallel section of the die starts. The shape of the deformation Δ can be characterised with these formulas (25):

$$\Delta = \frac{\text{average height of deformation zone } (\perp \text{ to drawing axis})}{\text{length of deformation zone } (// \text{ to drawing axis})} \quad (2.13)$$

$$\Delta \sim \frac{\alpha}{r} [1 + (1 - r)^{0.5}]^2 \sim \frac{4 \tan \alpha}{\ln(\frac{1}{1 - r})} \quad (2.14)$$

where low values for Δ imply small die angles and high cross-sectional reductions and high values mean large die angles and low reductions (25).

Moreover, the length of the deformation zone L_d , the die contact length L_c and the bearing length L_b are necessary for a thorough characterisation of the drawing geometry. The latter refers to the contact of the wire and the parallel die wall. The length parameters in the deformation zone are defined as follows (25):

$$L_d = \frac{d_0 - d_1}{2 \tan \alpha} \quad (2.15)$$

$$L_c = \frac{d_0 - d_1}{2 \sin \alpha} \quad (2.16)$$

where d_0 is the initial wire diameter and d_1 the drawn wire diameter.

Drawing Stress and Drawing Work

A drawing force F_d is necessary to pull the wire through the die. In continuous processes, another force in the opposite direction is exerted on the wire. This is called backpull force F_b and it results from low tension of the preceding capstan where the wire is spooled on (24).

The division of the drawing force F_d by the cross-sectional area of the drawn wire A_1 yields to the drawing stress σ_d . The backpull stress σ_b can be calculated respectively with the backpull force F_b and the initial cross-sectional area A_0 . Along the deformation zone the die pressure P acts on the wire. The stress σ_m shown in the centre of the deformation zone is an average of the stresses at the centreline and it is of great significance since its tensile values can cause fracture at the wire centre (25).

Drawing a wire with a length, l , and a drawing force, F_d , leads to work, W_d , as shown in equation (2.17). Rearranging this equation and dividing by the cross-sectional area leads to formula (2.18). Here, the left term represents the drawing stress, σ_d , and the right term is the work divided by volume or simply the work per unit volume, w_d . Hence, equation (2.18) can be simplified to equation (2.19). (25)

$$W_d = F_d \times l \quad (2.17)$$

$$\sigma_d = \frac{F_d}{A_1} = \frac{W_d}{l \times A_1} \quad (2.18)$$

$$\sigma_d = w_d \quad (2.19)$$

However, all terms in these formulations are determined and measured outside of the deformation zone and thus, they represent an external analytical view (25). A more useful way is to model the drawing stress from an internal perspective. This includes non-uniform or redundant work w_r , uniform work w_u and friction work w_f (25). Drawing stress can then be expressed as follows:

$$\sigma_d = w_u + w_r + w_f \quad (2.20)$$

Friction has a crucial contribution to the drawing stress. The frictional work in wire drawing and its analytical expression are discussed in more detail in section 2.3.1.

The work due to homogenous deformation of the wire is defined as uniform work (24,25). In this case, the interplay of die and wire are ignored and thence, a change in direction of metal flow and friction are not considered (25). A simple expression for uniform work in wire drawing can be defined, e.g.

$$w_u = \sigma_a \ln\left(\frac{A_0}{A_1}\right) = \sigma_a \ln\left(\frac{1}{1-r}\right) \quad (2.21)$$

where σ_a is the average flow stress in the wire (25). However, this formulation does not consider strain hardening which increases flow stress (25). To take strain hardening into account, the mean average flow can be calculated by the following,

$$\sigma_a = \frac{\sigma_i + \sigma_o}{2} \quad (2.22)$$

where σ_i and σ_o are the flow stresses at the inlet and the outlet respectively (24).

The redundant work is associated with the work resulting from shear deformations where the wire is in contact with the die wall and the continuity of velocity is interrupted (25). At the entry of the deformation zone the wire shears towards the centreline and it changes shear direction to the opposite when exiting the deformation zone, see Figure 2-5 (25). Since those shears even out and have no impact on wire geometry and reduction, this work is called redundant work (25). It is usually difficult to measure and model redundant work. A redundant work factor Φ , which shows the relation between uniform and redundant work, is commonly used and expressed as shown in equations (2.23) and (2.24), and an approximation to determine the redundant work factor is presented in equation (2.25) which shows a dependence on the shape of deformation Δ (25).

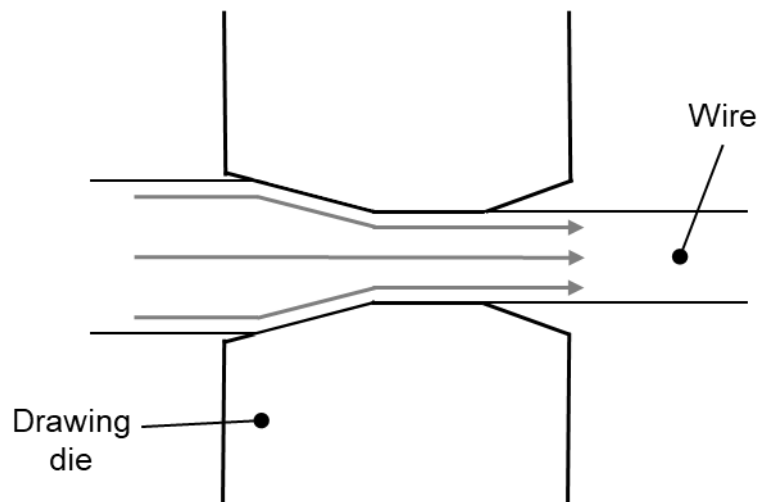


Figure 2-5: Material flow (gray arrows) in wire during drawing process adapted from (25) with permission from Elsevier.

$$\phi = \frac{w_u + w_r}{w_u} \quad (2.23)$$

$$w_r = (\phi - 1)w_u = (\phi - 1)\sigma_a \ln\left(\frac{1}{1-r}\right) \quad (2.24)$$

$$\phi \sim 0.8 + \frac{\Delta}{4.4} \quad (2.25)$$

2.2.2 Drawing Temperature

Wire drawing leads to a significant rise in temperature of the wire-die interface as well as the bulk wire and die material. Almost all work used to draw the wire is converted into heat and this effect leads to an increased temperature of die and wire (25). Since higher temperatures can cause changes in metallurgy (25), investigating the process temperatures is of great significance.

The die is only affected by a temperature rise from drawing in the start-up phase when the die is initially in a colder state. In industrial and steady-state drawing, the die already possesses a higher surface temperature, therefore, only a small amount of heat generated during drawing process flows into the die to keep it at the equilibrium temperature. Hence, it is commonly assumed that all heat generated in the wire drawing process is absorbed by the moving wire. (25)

The wire enters the drawing die with the inlet temperature T_0 . This inlet temperature can be higher than ambient temperature if the wire has already passed several consecutive drawing instances before and the resulting heat is not fully dissipated (25). After the wire is drawn through the die, a temperature gradient in radial direction usually exists, that is, the temperature on the wire surface is higher than in its centre due to frictional heating (25). However, the temperature within the wire bulk will quickly balance to an equilibrated temperature T_{eq} and is formulated as follows

$$T_{eq} \sim T_0 + \frac{\sigma_d}{C\rho} \quad (2.26)$$

where C is the specific heat capacity of the wire and ρ is the density of the wire (25). This expression includes the drawing stress since the work per volume unit is equal to the drawing stress as seen before in equation (2.19).

Similar to the drawing stress, the wire temperature increase includes contributions from uniform and redundant deformation as well as the friction (25). The highest prevailing temperature gradients are associated with frictional heating (25). Although they are only short-lived, those temperature gradients influence wire surface chemistry, lubrication, and metallurgical properties of the wire (25). El-Domiaty and Kassab (26) studied the effect of different process parameters on the temperature rise and energy consumption during wire drawing. They developed a first model which predicts the temperature rise with varying process and material parameters for ten different wire materials.

Other investigations on the effect of heating in high-speed wire drawing under hydrodynamic lubrication conditions were made by Lucca and Wright (27). They developed an analytical model to predict the resulting temperatures during wire drawing which considers heating of lubricant film and wire surface. Their work assumed a constant friction coefficient which is independent of temperature and pressure. In contrast, Vega *et al.* (28) studied the temperature effects in oil-lubricated wire drawing with an experimental approach and concluded a relationship between temperature and friction coefficient. Moreover, Haddi *et al.* (29) studied the effect of friction increase due to temperature rise on chevron cracks formation, and concluded that the damage zone increases with increase in friction. Chevron cracks are V-shaped surface fractures on the wire due to local sticking of wire to the die in boundary lubrication (25).

Temperature rise during wire drawing process has a profound effect on the tribological performance of the wire-die contact. If conventional lubrication mechanisms are assumed to exist in the wire-die contact, the rise in bulk wire and die temperature, and hence the inlet temperature, leads to a decrease of lubricant viscosity in the inlet to the wire-die contact which in turn leads to a reduction of film thickness and thus an increase in friction in the contact (30). The latter causes an increase in drawing stress (30). However, in the case of dry wire drawing with powdered soaps, the increase in temperature may also be a crucial step in the formation of the soap lubricating film, and hence, leads to lower friction and better surface protection.

2.2.3 Drawing Speeds

To increase productivity, wire drawing usually involves very high wire speeds up to 25 m/s (9) and in some cases even 50 m/s (31), whereby the drawing speed of the wire can be considered constant between subsequent drawing dies (25). During the drawing pass, however, the wire accelerates due to the cross-sectional area reduction and the related increase in wire length. A relationship between the inlet and outlet velocity and cross-sectional area are given in equations (2.27) and (2.28), assuming the volume of the wire remains constant,

$$u_0 \times A_0 = u_1 \times A_1 \quad (2.27)$$

$$\frac{A_0}{A_1} = \frac{u_1}{u_0} \quad (2.28)$$

where u_0 is the wire velocity before entering the die and u_1 is the velocity of the wire when it exits the die (25).

Drawing speed has an impact on various parameters. For instance, the drawing power is the product of drawing speed and drawing force (25). Hence, with increasing drawing speed, power consumption of the drawing process rises, assuming that the drawing force does not change. Moreover, there is a direct proportional correlation between drawing speed and strain rate, and the latter affects the wire's flow stress and as-drawn strength (25). Additionally, increasing drawing speeds reduces the time between two drawing dies and hence, the cooling time of the wire (25). As a result, the inlet drawing temperature of the subsequent drawing pass will then be higher and therefore, drawing temperature is rising steadily. Ultimately, drawing speed has a crucial effect on the prevailing lubrication mechanism as described in 2.1.3.

Tribology of wire-die interfaces plays an important role in wire drawing. It does not only influence the surface temperatures via friction, but it also contributes to the total drawing stress, wire quality, and die wear and failure. The next section is therefore focussed on friction and lubrication in wire drawing.

2.3 Tribology of Wire Drawing

At the wire-die contact, the wire is subjected to high frictional forces as described in 2.2.1 and shown in Figure 2-4. This leads to a high energy consumption as well as to potential damages to the wire and die surfaces. It is therefore crucial to understand the underlying friction and lubrication conditions in wire drawing to save energy and improve wire quality. However, literature in the field of tribology, friction and lubrication of wire drawing appears to be extremely limited compared to the wider wire drawing research. This is illustrated in Figure 2-6. This section describes the noteworthy previous studies in this field.

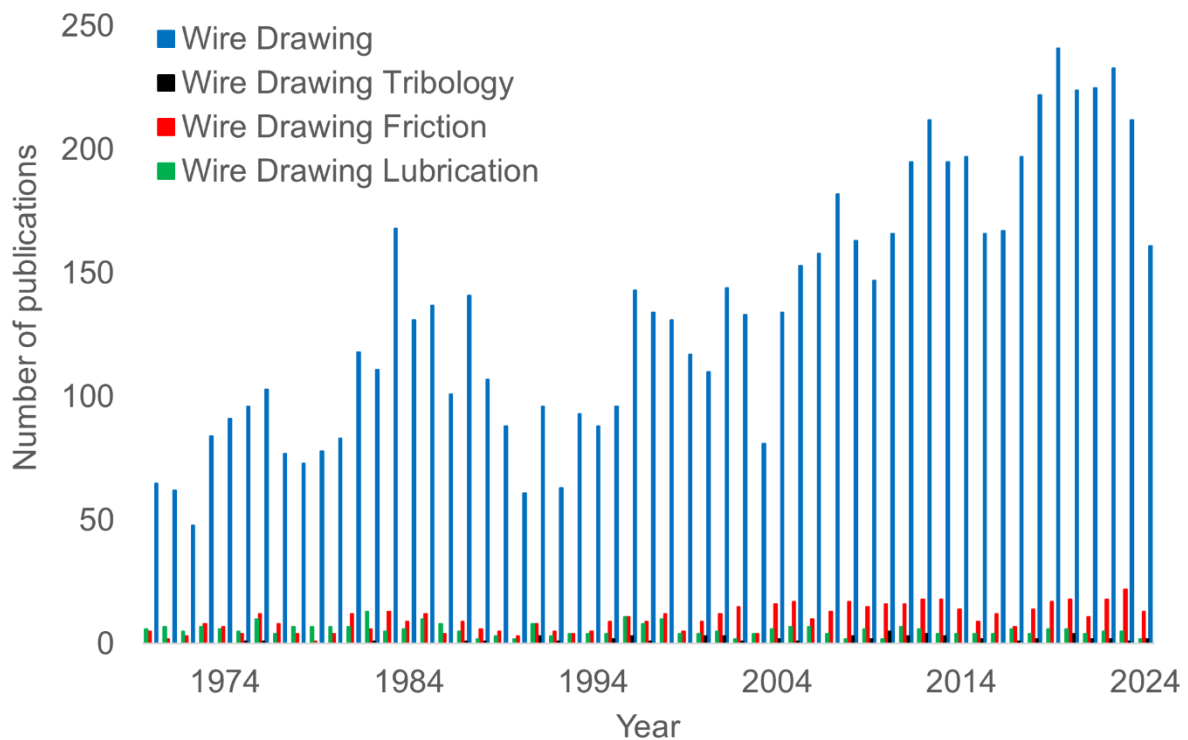


Figure 2-6: Publications related to the tribology of wire drawing over the last 50 years (Results obtained on Scopus (32) for search terms “wire drawing”, “wire drawing tribology”, “wire drawing friction”, “wire drawing lubrication”).

2.3.1 Friction in Wire Drawing

During the drawing process, a pressure P from the drawing die is exerted on the wire surface as shown in Figure 2-4. This normal pressure and the sliding of the wire at the die wall lead to shear stresses in the opposite direction to the sliding direction of the

wire. The Coulomb friction coefficient (COF) can then also be described as the following (24):

$$COF = \frac{\tau}{P} \quad (2.29)$$

In wire drawing, the die pressure is a term which reflects both redundant and uniform work in the deformation area and can be approximated as the product of redundant work factor ϕ and average flow stress σ_a (25). The frictional work, which contributes to the drawing stress, can be derived through the multiplication of frictional stress and contacting area and the subsequent division by the reduced cross-sectional area (25). Hence, the frictional work per unit volume can be expressed as follows (25):

$$w_f = COF \cdot \cot\alpha \phi \sigma_a \ln\left(\frac{1}{1-r}\right) \sim \frac{4COF \cdot \phi \sigma_a}{\Delta} \quad (2.30)$$

2.3.2 Measurement of Friction in Wire Drawing

To determine the magnitude of friction between wire and die, it is important to perform measurements during wire drawing. However, determining the frictional force or the friction coefficient directly during the process of drawing is difficult. Hereafter, some methods to estimate the friction coefficient in the wire-die contact are described.

In-direct Friction Measurements – Split-Die Method

One way to approximately measure friction in wire drawing is the Split-die technique developed by Wistreich (33). This method includes a wire drawing die that is split in half longitudinally. When drawing the wire through the split-die, the drawing stress and the force to separate the two halves of the die are measured simultaneously (33). From this, the mean coefficient of friction and the mean die pressure P_m can be calculated as follows,

$$COF = \tan\left(\frac{F_d}{\pi F_s} - \alpha\right) \quad (2.31)$$

$$P_m = \frac{\pi \cdot F_s}{(A_0 - A_1)(\cot\alpha - COF)} \quad (2.32)$$

where F_d is the drawing force, F_s the separating force, and α is the semi-die angle (33). However, a drawback of this approach is that some parts of the lubricant pass through the die split (25), and the metal wire is partly squeezed out into the gap (34), both of which can influence the measured friction coefficient. Moreover, this technique requires modifications of the wire drawing machine to implement suitable instrumentation.

Direct Friction Measurements – Upsetting-Sliding Test

A more direct method to measure friction in a pure sliding motion is the upsetting-sliding test (7,35). In this technique, an indenter made of die material moves upwards along a specimen made of wire material, and thereby deforms it plastically. The normal and tangential forces are measured, and friction can be calculated (35). In this test set-up the specimen (“wire”) goes through different baths to obtain a soaped coating. The set-up also includes a lubricating nozzle to provide additional oil during testing. Despite the ability to measure friction directly in a sliding motion, this method was limited to low sliding speeds of 0.75 m/s. Furthermore, a pre-coating of the specimen with the lubricant was necessary which has to be sufficient enough to remain on the specimen during the upward sliding movement.

Reversed Engineering Methods – Optical Die Angle Method

Evans and Avitzur (36) derive the friction coefficient from the identification of the optimal die angle. This technique includes several drawing tests with different die angles assuming constant drawing conditions, e.g. cross-sectional reduction, friction and flow stress. During wire drawing, the drawing stress is measured. By plotting the drawing stress against the die angle, the optimal die angle can be identified. The frictional coefficient can then be calculated analytically by formulas depending on the drawing stress and the optimal die angle. However, Vega *et al.* (28) pointed out that the friction coefficient during wire drawing is not constant but is related to the temperature. Hence, this technique is not applicable for high speeds which lead to high temperature rise in the wire-die contact.

Reversed Engineering Methods – Thermo-mechanical Model

Felder *et al.* (37) developed a technique based on a thermo-mechanical model with which both the friction and the plastic deformation can be measured concurrently. This

approach allows the identification of the friction stress and the stress strain curve.

During drawing with calcium and sodium soaps, the drawing force and the die temperatures are measured (37). For the latter, two thermocouples are mounted in the drawing die, where one (T_2) is close to the wire-die contact, and the other one (T_1) is located at the interface of the die kernel and the die ring as shown in Figure 2-7 (37). The location of thermocouples is important but, regardless, this cannot provide the direct measurement of the in-contact temperatures but only a rough estimate.

This thermocouple set-up was used to identify a linear correlation between T_1 and T_2 which supports the assumption that average die heating is proportional to maximum die heating (37). Since the latter also includes drawing speed and friction stress, they assume that speed and friction changes only lead to a shift in amplitude of the distribution of die temperature (37).

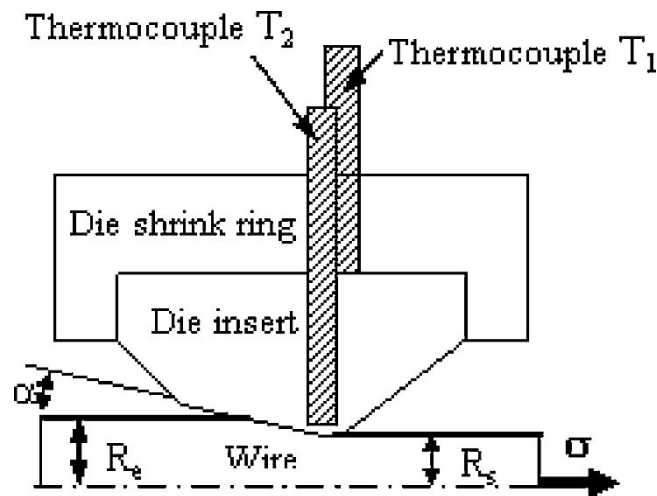


Figure 2-7: Location of thermocouples in experimental set-up reprinted from (37) with permission from Elsevier.

According to their numerical model, the drawing stress σ_d and the die temperature are correlating, i.e. (37)

$$\sigma_d = W_{pl} \left(1 - \frac{\xi}{\rho C \varphi(u)} \right) + \left(\frac{\beta \xi}{\varphi(u)} \right) \Delta T_{max} \quad (2.33)$$

where W_{pl} is the plastic deformation, ρ is the wire density, C is the wire specific heat capacity, β is the ratio between the maximal die heating and measured die heating, ΔT_{max} is the maximum surface temperature rise (die or wire) and ξ and $\varphi(u)$ are defined as follows (37)

$$\xi = \ln \lambda \cot \alpha + \frac{2L_b}{R_1} \quad (2.34)$$

$$\varphi(u) = \sqrt{\frac{3}{2}} \frac{\sqrt{uL_t}}{b} \left(1 + \frac{3}{2} \frac{k_d}{bL_t} \sqrt{\frac{L_t}{u}} \right)^{-1} \quad (2.35)$$

where λ is the elongation ratio, α is the die semi-angle, L_b is the die bearing length, R_1 is the exit wire radius, u is the wire axial velocity, L_t is the total length of the wire-die contact, b is the wire thermal diffusivity and k_d is the die thermal conductivity.

From this, the work for plastic deformation W_{pl} can be determined from equation (2.33) and the friction stress τ can be calculated from the following (37):

$$\tau = \frac{\sigma - W_{pl}}{\xi} \quad (2.36)$$

The comparison of the experimental results of temperature rises at the die-wire interface (ΔT_2) showed rather good agreement with results from numerical simulation (37,38). However, the measured outcomes are highly sensitive to the exact locations of the thermocouples, and the thermocouple at the wire-die interface averages the temperature over a large part of the deformation zone (37).

Further analytical and reversed engineering methods exist to estimate friction in wire drawing, e.g. (7,39,40). However, although these approximate approaches may be helpful for rough evaluation of the process on a factory floor, they suffer from multiple deficiencies and are less valuable in providing understanding of the tribological mechanisms at play in the wire-die contact (41). It is also difficult to do systematic studies of frictional behaviour of the wire-die contact using an actual wire drawing process since

the tribological conditions in the wire-die contact cannot be controlled (41).

2.3.3 Lubrication in Wire Drawing

Lubrication in wire drawing is highly relevant as it reduces friction, die wear as well as heat generation and ensures process stability (22,24). Beside the reduction of friction at the wire-die interface, lubrication can also perform a cooling function and needs to be resistant to high pressures and high temperatures (22). Commonly used lubricants in wire drawing are fluids, e.g. emulsions, or solids, e.g. powdered soaps. The former is used in wet wire drawing where the wire is usually completely immersed in the liquid. In dry wire drawing, which is the preferred method for drawing relatively thick wires of around 1 mm or larger in diameter (42) due to better performance and lower energy consumption², the wire passes through a container with a solid, dry soap lubricant before entering the die.

There is little understanding of the actual lubrication mechanism with soaps and even the lubrication regime in the die-wire contact is unknown. Important process parameters such as the types of soap for different speeds and wire reduction ratios, optimal speeds for required wire quality etc., are currently all based on the operator know-how and past experience on the manufacturing floor. Anecdotally, it also appears that this know-how varies between different manufacturing sites. This obviously acts as an obstacle to the implementation of potential improvements to the dry wire drawing process for reduced energy consumption and improved wire quality.

Wet wire drawing

Research into wire drawing tribology is very limited and one of the first studies on lubrication behaviour during wire drawing is due to Christopherson and Naylor (43). In conventional wire drawing it is believed that mixed and boundary lubrication conditions are most likely present (44,45). According to the work of Christopherson and Naylor (43), however, a hydrodynamic fluid film could be promoted with oils if there is enough pressure provided at the drawing die inlet which increases the amount of lubricant passing through the drawing die. In order to generate the required pressure for

² Implicit industry know-how, personal communication by Stijn de Pauw and Hendrik Van Hoecke, Bekaert

hydrodynamic lubrication, they developed a tube system connected to the drawing die and performed first experiments with copper and mild steel wires with an oil as a lubricant. They also formulated first theoretical explanations for their experimental outcomes. However, their equations were expressed with dependent parameters such as the developed pressure and the quantity of the lubricant flow (46). Based on the work of Christopherson and Naylor (43), Tattersall (46) developed a theory which takes all regions up to the deformation zone into account and restated the equations by making the developed pressure and the quantity of lubricant flowing independent. This theory was compared to the experimental results of Christopherson and Naylor, and with some modifications, it agreed well for both the values of pressure and lubricant flow. Importantly from the standpoint of this thesis, Tattersall investigated the applicability of this theory to dry soap lubrication and emphasised that the rheological properties of the soaps under high shear rates and high pressures needed to be understood first before any such theory could be applied to dry wire drawing. In the work of Osterle and Dixon (44), the theory of Christopherson and Naylor (43) was adapted again by including temperature effects in the tube and plastic flow effects of the wire in the die region using a viscous lubricant.

Another theoretical approach was given by Bedi (47) who used Reynolds' equation to calculate film thickness and viscous friction coefficient with a Newtonian fluid in wet wire drawing. Bedi's model demonstrated the potential of full hydrodynamic lubrication during high-speed wire drawing with a conical die. It was also deduced that the prevailing friction coefficient varies along the die. However, the influence of temperature, strain rate and pressure effects were not considered in Bedi's model.

An approach to further reduce friction in fluid lubricated wire-die contacts was used by Su and Marek (48). They performed electromechanical tests with emulsions to analyse the effect of applied voltage on friction. In their lab tests, a tungsten carbide flat was loaded against a rotating copper cylinder, and a potentiostat was used to control the potential of the copper. They concluded that this electrode control method was able to reduce friction by improving lubrication and preventing direct contact between die and wire. However, this method requires major adaptations to the drawing setup.

Some experimental efforts were done to directly observe the lubricant film in a wire

drawing machine (49,50). Nanao *et al.* (49) observed the formation of an oil film between the wire and the die under static conditions after drawing by using a die with a diamond window and micro-Fourier transform infrared spectroscopy (FTIR). They measured the infrared spectra of the oil film in the contact zone after drawing and estimated the film thickness through a calibration curve from an IR spectra of a known film thickness. Based on that method, the technique developed by Hoshi *et al.* (50) allowed micro FTIR measurements of the film directly during wire drawing. Their in-situ observation also helped in obtaining the concentration of the lubricant additives. It was furthermore found that the additives used in their study reacted with the brass-coated steel wire and formed metal soap which led to decreased drawing force and better surface finish. However, these studies considered low drawing speeds of only 0.2 m/s, and could only infer film thickness from a known reference IR spectrum. A method to obtain film thickness directly from in-situ measurements of the wire-die contact does not exist yet.

Dry wire drawing

In terms of dry wire drawing with metallic soaps, first studies identified the significance of soap rheology (51,52). Montmitonnet *et al.* (52) derived a thermopseudoplastic model which considers thermal and non-Newtonian effects of the lubricant. Studies have shown that soaps undergo a phase transition at elevated temperatures (53), and exhibit plastic behaviour at low temperatures and non-Newtonian viscous behaviour at higher temperatures (51). Including this rheological information of soaps improves film thickness calculations in wire drawing (51,52). For example, Montmitonnet *et al.* (52) found a linear dependence of viscosity on both temperature and shear rate, e.g. for an industrial calcium soap, which proved a pseudoplastic behaviour of this soap:

$$\eta = \eta_0 \exp(\gamma p - \delta T_s) \dot{\gamma}^{m-1} \quad (2.37)$$

where η and η_0 are the viscosity and base viscosity respectively, γ is the coefficient of piezoviscosity, p is the pressure in the lubricant film, δ is the coefficient of thermoviscosity, T_s is the soap temperature, $\dot{\gamma}$ is the shear rate, and m is the coefficient of pseudoplasticity of the soap. Using this non-Newtonian behaviour and classic hydrodynamic analysis, e.g. solving Reynolds' equation, they derived a model to measure film thickness over the whole wire-die contact. Their model predicted high film thicknesses

in the range of tens of μm depending on the values of the coefficients of pseudoplasticity m and thermoviscosity δ . However, the validity of the assumption of hydrodynamic lubrication of dry soaps is highly questionable.

Levrau (54) also studied the lubrication mechanisms during wire drawing with dry soaps in a real wire drawing machine. Levrau's work involved analyses of the residual weight of soap film, the drawing stress, the die temperature as well as optical analysis of the wire surface. By identifying distinct areas on the wire surfaces after drawing, such as areas of bare metal, coating or residual soap film, using SEM-EDX and measurements of residual weight of wires, Levrau concluded mostly mixed lubrication conditions to prevail during conducted wire drawing tests. It was shown that lubrication was positively influenced by high roughness of the die and wire, a low die angle and the use of a pressure device.

Another factor that is believed to affect the lubrication behaviour in dry wire drawing is the particle size of the powdered soap at the initial stage of drawing (55). Some studies suggest that smaller particles lead to a fully formed lubricating layer on the wire, whereas large particles retard the full film formation (55).

To optimise wire quality and die life in dry wire drawing, some process monitoring methods were developed using for example a CCD camera (56), vibration sensing (57), or thermal imaging cameras (58) to detect deficiencies in the process. For instance in the work of Larsson *et al.* (58), thermal images of the wire as it exits the drawing die were studied to uncover changes in lubrication conditions. Poor lubrication conditions, e.g. lack of lubricant on wire surface, or wire surface damages resulted in different thermal signals compared to wire with good lubrication conditions, e.g. wire fully covered in lubricant. Their results showed good similarities with previously used process monitoring methods observing drawing force measurements, but the applicability still needs to be evaluated in terms of endurance and accuracy (58).

Huo (59) studied the lubrication efficiency in wire drawing with powdered soaps experimentally and using finite element methods. Huo's research also assumed that the soap would liquify due to frictional heating in the contact and hydrodynamic lubrication was realised in the wire-die contact. There is no direct evidence to support either of

these assumptions. The work also highlighted that drawing stress varies with drawing speed due to changes in friction and lubrication conditions of different soaps.

An alternative to dry wire drawing with powdered soaps was introduced by Eickemeyer *et al.* (60). They coated wires prior to drawing with an aqueous dispersion which formed a solid soap film after drying. It was suggested that hydrodynamic pressure was promoted with this soap lubrication which in turn led to low friction. Although this method entails a pre-coating process, it delivers some environmental benefits such as reduced soap dusting and less use of lubricant carriers (61).

2.4 Critical Summary of Literature and Objectives of Thesis

The existing research pertinent to the scope of the thesis has been presented. Several experimental research and numerical studies of friction and lubrication conditions in wet wire drawing with fluid lubricants were carried out. In contrast, friction and lubrication in dry wire drawing with powdered soaps, which is a common method to produce steel wires, are not yet fully understood. The likely reasons for this are the complexities in the tribological behaviour of powdered soaps and the complex contact conditions in the wire-die interface. Wire drawing involves high pressures due to the plastic deformation of the wire and very high sliding speeds needed for increased productivity. Other than the general belief that soaps exhibit a plastic behaviour at low temperatures and non-Newtonian viscous behaviour at high temperatures, the rheological behaviour of powdered soaps largely remains a mystery. The situation is not aided by the fact that there appear to be no direct observations of the soap films either in the die-wire contact itself or in an appropriate lab set-up.

Literature concerning the tribological interactions in wire drawing is extremely limited. Research existing in this field focuses almost exclusively on wet wire drawing with emulsions or oils; dry wire drawing with powdered soap has barely been studied.

Commonly, the understanding derived from these studies on wet wire drawing and fluid lubrication theories are extrapolated to powdered soap lubrication in dry wire drawing. This, for example, leads to the assumption of a hydrodynamic or quasi-hydrodynamic lubrication regime with powdered soaps with no supporting evidence of this. It is not

clear, however, if standard hydrodynamic film behaviour does apply to soaps as the solid nature of these soaps exhibits different rheological behaviour than standard emulsions or oils. In addition to these theories, important process parameters in wire drawing with soaps are based on the operator know-how and industrial experience rather than a thorough understanding of the actual lubrication mechanisms and lubrication regimes in dry wire drawing. Increased knowledge of the lubrication behaviour of powdered soaps could lead to potential improvements to wire surface quality and energy reductions in wire drawing. However, the nature of the soap films or their formation mechanisms are still unclear since these films have not yet been observed directly.

In terms of friction measurements, several studies have devised ingenious ways to estimate the friction coefficient in the wire-die contact during the actual wire drawing process. None of these methods measure friction force directly but instead, they infer friction from some other measured parameter, such as temperature for example. Importantly, although measuring friction on the actual wire drawing set-up has the obvious advantage of accounting for realistic conditions, it does not allow for a systematic investigation of the frictional behaviour of powdered soaps since the wire-die contact conditions cannot be directly controlled. Such systematic study is necessary to advance our understanding of basic frictional mechanism at play, as well as to simply quantify the friction coefficient with different powdered soaps and contact conditions, which can be used in process optimisation.

Metal soap powders for dry wire drawing are widely and successfully used in the wire drawing industry but their frictional and film forming behaviour is unclear. This hinders improvements in wire drawing processes that are necessary for improvements in wire quality and, perhaps more significantly, for the reduction in energy consumption during the wire drawing process, effect of which can be very significant given the high energy intensity of the process. The present thesis aims to help address this by systematically studying the friction and film behaviour of powdered soaps under controlled conditions and hence providing new quantitative data on friction that can be used in practice, as well as new insights into the frictional and film forming mechanisms of dry soaps.

In view of the gaps identified in the literature, this research attempts to answer the following specific questions:

-
- Is it possible to measure friction directly with dry powdered soaps in conditions pertinent to the wire-die contact in a systematic and controlled manner?
 - How do process parameters such as shear rates (SRR), speeds, pressures, materials, soap formulations and particle sizes affect friction behaviour?
 - What is the nature of soap lubricating films and how do soap powders entrain into the contact?
 - What effect does temperature have on soap films?

This is achieved by completing the following research tasks:

- Tests on a ball-on-disc tribometer to study the effect of high speeds, pressures and sliding on friction similar to the harsh conditions found at the wire-die interface.
- Optical and chemical analysis of lubricating layers on surfaces post friction test to gain insights into the nature of lubricating layers.
- Direct observation of powder entrainment and film formation using a ball-on-disc optical set-up.
- Analysis of temperature effects using analytical methods and differential scanning calorimetry.
- Comparison of findings from lab experiments to tests conducted on a real wire drawing machine.
- In light of all findings, new insights into the nature of soap films and the lubrication mechanisms of soap powders.

3 Research Methods and Experimental Equipment

This chapter starts by briefly describing the general research approach taken in this work in relation to the knowledge gaps identified in Chapter 2. Following this, the experimental techniques used to explore frictional performance and lubrication behaviour of dry wire drawing soaps are explained here.

3.1 General Research Methodology

The general objective of this thesis is to study friction and lubrication in dry wire drawing as described in chapter 1.2, and therefore, answer the gaps as described in chapter 2.4. A major issue with existing dry wire drawing soap research is the lack of a systematic and controlled study to directly measure friction and observe soap film formation.

To address this matter, in this work friction was studied in a ball-on-disc lab tribometer, namely the extreme-traction machine (ETM), with which it is possible to test soap powders under controlled conditions including high pressures representative of real wire drawing processes. The rig also allows for the control of slide-roll-ratios, sliding speeds and test temperatures. Although a ball-on-disc contact exists in the ETM, contrary to the conformal contact in the real application, this machine can directly measure friction with dry powdered soaps under the severe conditions very similar to wire drawing conditions. Material pairs, temperatures and contact pressures were chosen to mimic the tribological conditions in the wire-die contact. The ETM machine can reach sliding speeds up to 4 m/s which is higher than most friction measurements shown in past literature (Chapter 2.3.2) and it depicts the lower spectrum of industrial wire drawing speeds. Alternative tribometers such as the PCS HFRR (ball-on-plate reciprocating friction) machine or a pin-on-disc machine were explored to reproduce a pure sliding motion, but applicability was limited due to difficulties in soap re-entrainment and lower pressures compared to the ETM machine. To confirm the applicability of the ETM observations to real dry wire drawing, the ETM results are compared qualitatively to real wire drawing tests performed at a test bench by the sponsors of this project. This

includes comparison of residual soap films on ETM specimens and those on drawn wires. In addition, the trends apparent in ETM results are related to practical ‘know-how’ in terms of behaviour of different soaps reported by the employees of the sponsoring company.

To better understand the lubrication behaviour of the tested soap powders, and the formation mechanisms and the nature of soap lubricating films, an optical ball-on-disc machine (EHD-HS) was used. This machine allows you to directly look into the contact and observe the entrainment of soap particles as well as any film formation. Again, the optical EHD rig allows for control of test conditions. The formed soap films are also analysed post-test using optical techniques, such as confocal and SEM-EDX microscopy, and Raman spectroscopy.

3.2 Friction Measurements

The frictional behaviour of soaps is studied using a ball-on-disc tribometer. The machine used and the material properties of the specimens, as well as the test conditions and procedures are explained in the following.

3.2.1 ETM Rig

To study friction with dry powdered soaps, the ETM by PCS Instruments (62) is used. This ball-on-disc tribometer is capable of mimicking the severe conditions in wire drawing as it is capable of high loads up to 1,500 N, equivalent to 4.0 GPa maximum Hertzian pressure in a tungsten carbide (WC) ball on steel disc contact. In the ETM, a $\frac{3}{4}$ inch diameter ball is loaded and rubbed against a flat disc in a rolling-sliding contact. Ball and disc are driven independently, so that slide-roll-ratio (SRR) can be varied between pure rolling and pure sliding, the latter being representative of the wire-die contact. The SRR in this work is defined as the ratio between sliding speed u_s and entrainment speed u_e as defined in (3.1), where u_{disc} and u_{ball} are the surface speeds of the disc and the ball respectively (41). Hence, an SRR of 0% represents pure rolling and an SRR of 200% pure sliding (41).

$$SRR = \frac{u_s}{u_e} = \frac{u_{disc} - u_{ball}}{\frac{1}{2}(u_{disc} + u_{ball})} \quad (3.1)$$

The ETM machine is shown in Figure 3-1. To run experiments with soap powders, a PTFE scoop was included in the ETM, similar to that previously used in grease studies on the MTM, e.g. (63). The scoop is mounted in the test chamber so that it pushes the soap powder back onto the rubbing track during the experiment and therefore, ensures a continuous supply of soap powder at the inlet and prevents starvation in the contact which would have otherwise occurred with powdered lubricants. Therefore, the scoop acts in a similar manner to the funnel at the front of the die in the actual wire drawing set-up as described above.

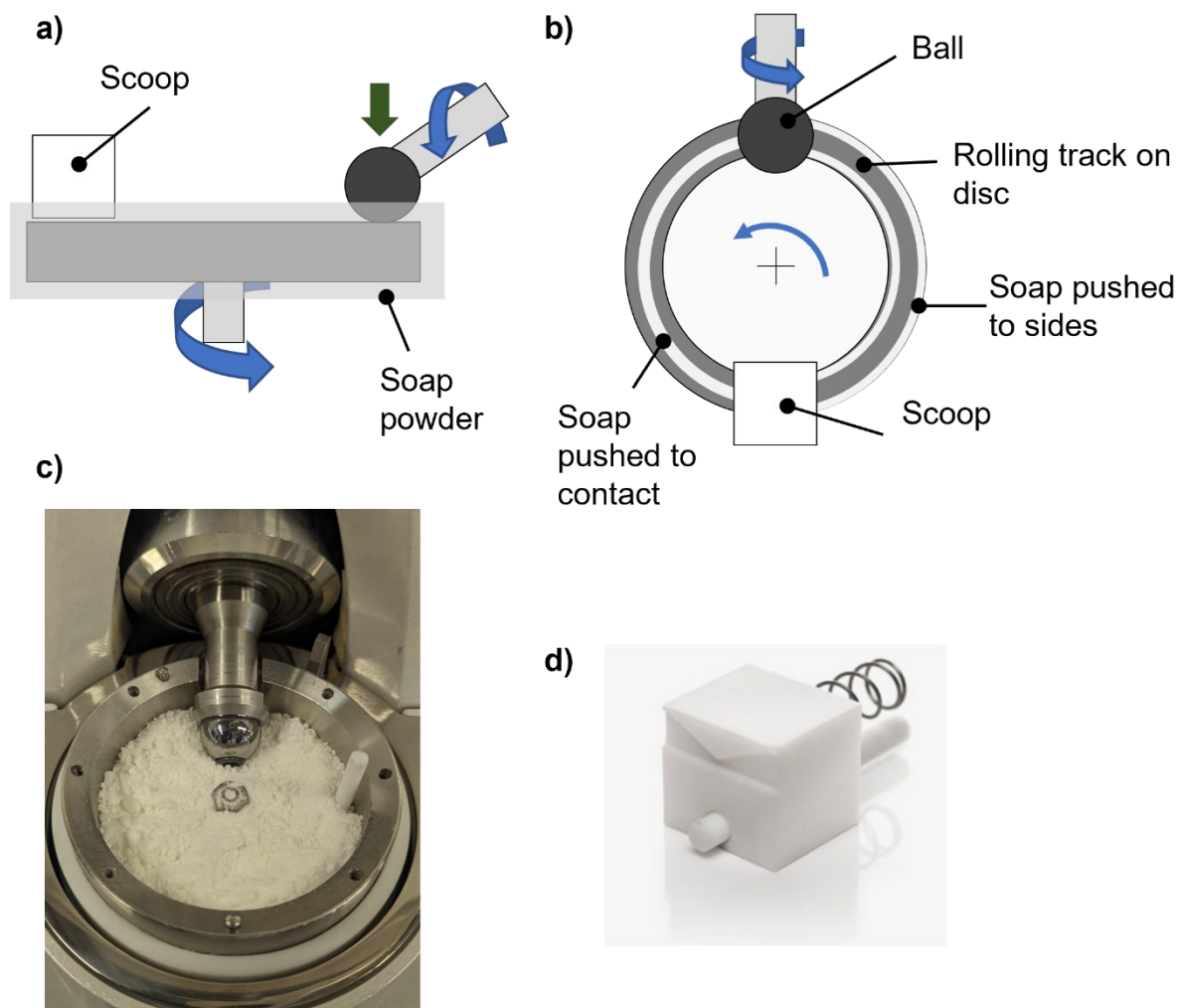


Figure 3-1: a) A diagram of the ball-on-disc ETM rig including the soap scoop, b) principle of soap scoop, c) an example photograph of the ETM pot with mounted specimens and filled with sodium soap, d) a photograph of the scoop (64) reproduced with the permission of PCS Instruments.

3.2.2 Material Properties of ETM Specimens

The ETM disc specimens were custom made so that the actual wire drawing process could be mimicked. A low carbon steel C15E was employed for the disc specimens. This is a steel grade that is very similar in composition to typical wire drawing steels, for example C15D. The steel disc, however, was case-carburised to increase the surface hardness of the discs and therefore, avoid plastic deformations in the experiments which would affect contact conditions and hence measured friction. Some C15E specimens in their initial annealed state were also tested to study the effect of plastic deformations during rubbing tests on friction.

Since wire drawing dies are usually made of WC, the ETM ball specimens in these tests were also made of WC. The properties of the ETM ball and disc specimens are listed in Table 1.

Table 1: Properties of the ETM ball and disc specimens

	Ball	Disc
Materials	WC	C15E steel case carburised (steel similar to that used for many wires)
Young's Modulus, E [GPa]	600	200
Poisson's Ratio, ν [-]	0.293	0.28
Diameter, d [mm]	19.05	46.00 overall (mean track diameter 41.60)
Roughness, Rq [nm]	~ 15	~ 15
Hardness / HV30	1600 $\pm$ 30	703 $\pm$ 10

3.2.3 ETM Test Conditions and Procedure

Since the wire drawing process involves very high contact pressures in the GPa range (7,8) which is needed to plastically deform the wire, and very high sliding speeds of up to 25 m/s (9), severe test conditions were chosen to better represent the harsh contact conditions in the wire-die contact. Maximum Hertzian pressures of 2.0, 2.5 and 3.0 GPa and a pot temperature of 40°C were used in the ETM tests, similar to typical conditions found in real wire drawing. The ETM contact conditions are shown in Table 3. Friction trends from the lab tests under these conditions and trends observed on a

real wire drawing machine are compared in chapter 7.

To study the frictional behaviour of the soaps, Stribeck curves, traction curves and long-term tests were conducted with the test conditions shown in Table 2. Unlike the real wire-die contact, the ETM tests were not conducted in pure sliding (SRR = 200%) but, a maximum SRR of 180% was set, i.e. close to pure sliding. This was done for better control of test conditions, namely, to ensure that any wear is spread over a larger area of the ball specimen so that its impact on contact conditions is minimised. A SRR of 180% is close enough to pure sliding to account for any effect of high sliding speeds. A maximum entrainment speed of 2 m/s was tested for a SRR of 180% ($u_s = 3.6$ m/s). Traction tests were performed over a range of SRRs from 0% to 180%, and Stribeck tests over a range of entrainment speeds from 0 to 2 m/s. Long-term friction tests were conducted at 2.0, 2.5, 3.0 GPa and a fixed entrainment speed of 1.8 m/s and SRR of 180%. A fresh set of specimens was used for each test.

Table 2: Test conditions used in the ETM friction tests

Maximum Hertzian pressure [GPa]	2.0, 2.5, 3.0
Test temperature [°C]	40
SRR	180% (+ Traction curves with SRR = 0% – 180%)
Entrainment speed (u_e) [m/s]	2 (+Stribeck curves with $u_e = 0 - 2$)

Table 3: Contact size of ETM ball-disc contact at three test pressures

$P_{\max}$ [GPa]	P_{mean} [GPa]	a [μm]	W [N]
2.0	1.33	183.5	141.0
2.5	1.67	229.3	275.4
3.0	2.00	275.2	475.9

Prior to the tests, the specimens were cleaned with toluene or heptane followed by isopropanol in an ultrasonic bath. The ETM test chamber, as well as all mounting parts and tools were rinsed, wiped and cleaned with toluene or heptane followed by isopropanol. The clean specimens were then mounted in the test chamber of the ETM, and the experiments were performed under specified test conditions.

The surface roughness of each disc and ball specimen was measured before and after the tests using a stylus profilometer and a confocal microscope as mentioned above. To understand the nature of the deposited surface layers in the rubbing track, the rubbing tracks of the specimens were analysed post-test using Raman spectroscopy as well as SEM-EDX.

Lastly, the confocal microscope was also used to measure the thickness of any deposited soap films on the discs using the following procedure (41): 1) a short segment of the rubbing track was wiped with isopropanol to remove the formed film, 2) the disc was inspected under the confocal microscope and the height difference between the surface in the cleaned area and the top of the untouched film layer was measured, 3) a total of 20 measurements (over several areas) per each disc were made in this way and the average of these was calculated. The procedure is illustrated in Figure 3-2.

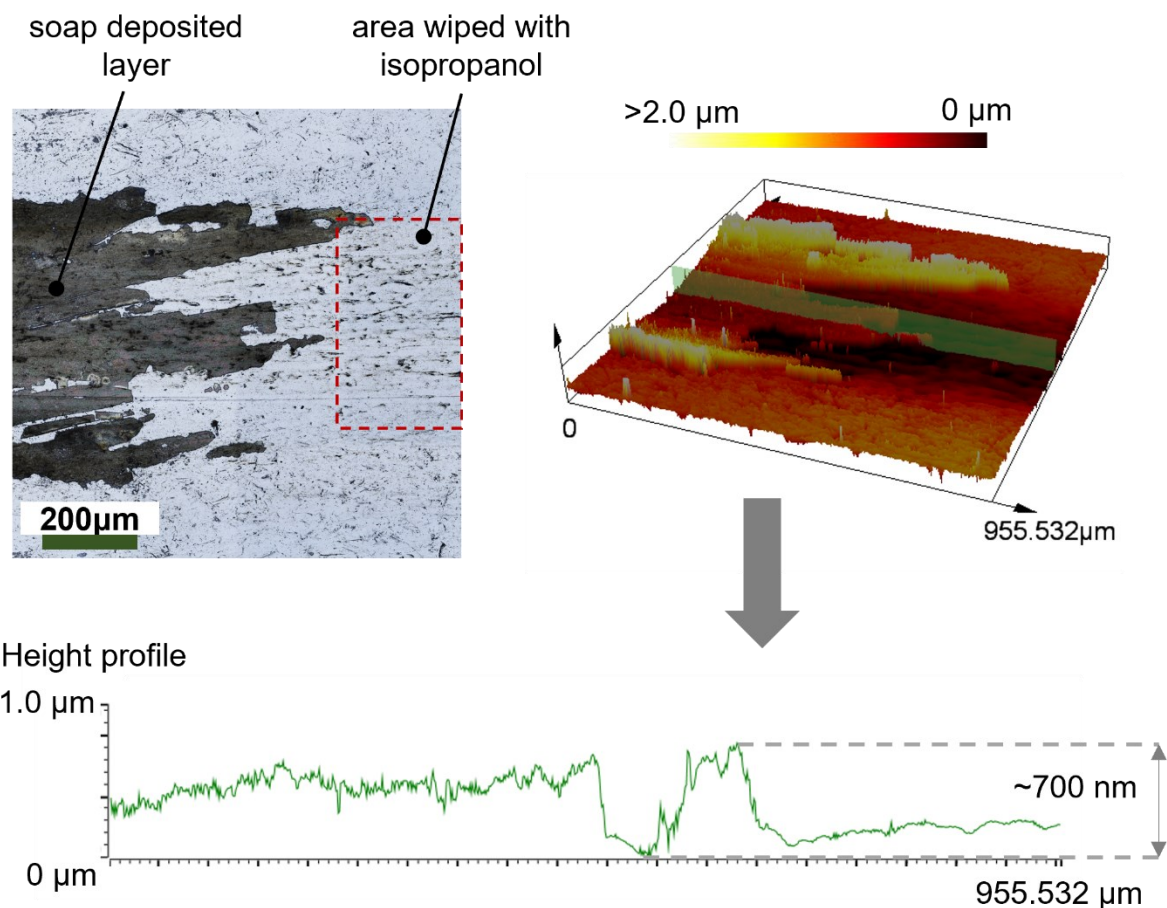


Figure 3-2: Procedure of measuring film thickness of residual film on rubbing track

3.3 *Film Build-Up Observations*

The build-up of the soap films in the contact was directly observed using an optical ball-on-disc machine. The optical set-up, the material properties, and the test conditions and procedures are described here.

3.3.1 EHD-HS Rig

An optical ball-on-disc rig is used to observe film formation directly in the contact. For this, the high speed EHD machine (EHD-HS) by PCS Instruments (65) is used which allows for entrainment (and sliding) speeds of up to 20 m/s and therefore, better represents the speeds in real wire-die contacts during the drawing process. The rig is very similar to the standard PCS optical EHD rig (66) other than the high speeds and its ability to measure friction in ball-disc contacts more accurately using a reaction torque sensor. The standard set-up in the EHD-HS machine uses optical interferometry (67) to measure film thickness in the contact of a steel ball and a silica chromium coated glass disc. However, in this study the EHD-HS was mainly used to observe the contact and the lubrication behaviour of the soaps which was done with the camera set-up shown in Figure 3-3. A steel ball is loaded against a transparent disc. Ball and disc are driven by separate motors, so that different SRRs and high sliding can be achieved. The contact is illuminated by a white light source so that it can be seen via a camera. Common objectives used have a magnification of 5x, 10x or 20x. The pot where the ball is mounted is filled with soap powder. The main use of this machine was to give implications on potential lubrication mechanisms at play by direct observations of soap entrainment and film formation. Given the different material pairs, the contact pressure is lower than in the ETM tests and real wire drawing but friction trends showed similar behaviour to those seen in the ETM friction results.

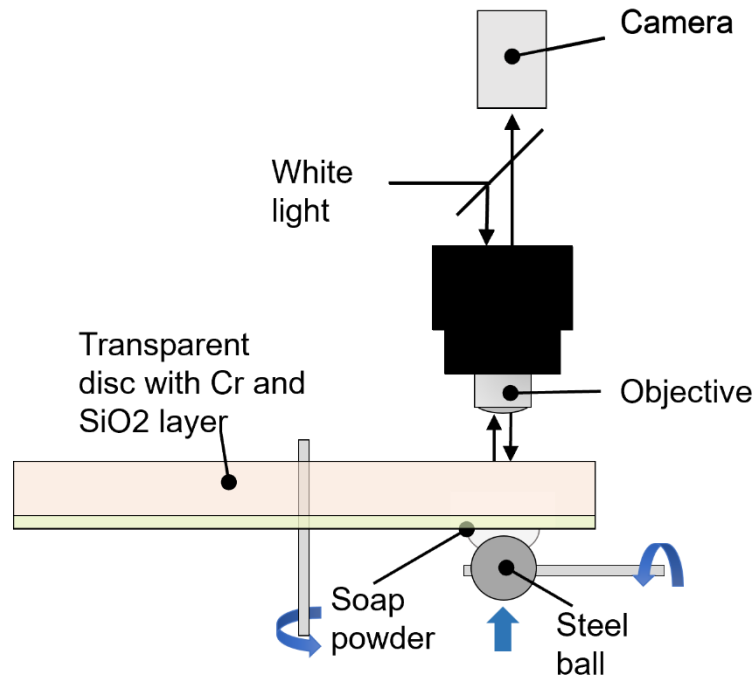


Figure 3-3: A diagram of the EHD/EHD-HS using the camera-set-up used here to observe soap films

3.3.2 Material Properties of EHD Specimens

In the EHD set-up, a transparent disc is required to look into the contact. Therefore, the discs are made of glass for lower pressures or sapphire for higher pressures. The transparent disc is coated with a chromium silica layer.

A standard bearing steel AISI 52100 was used for the balls. Properties of the EHD specimens are listed in Table 4.

Table 4: Properties of the EHD ball and disc specimens

	Ball	Disc
Materials	AISI 52100 bearing steel	Glass / Sapphire
Young's Modulus, E [GPa]	207	78 / 400
Poisson's Ratio, ν [-]	0.29	0.2 / 0.3
Roughness, R_q [nm]	~15	< 1

3.3.3 EHD Test Conditions and Procedure

Given the use of glass and sapphire discs, lower pressures were used in the EHD set-up compared to the ETM set-up. The tests were run under maximum Hertzian pressures of 0.5 GPa and 1.0 GPa with the glass disc and sapphire disc respectively. The same high SRRs of 180% were also tested in the EHD set-up for some tests. But given that high sliding quickly destroys the silica coated disc surfaces, the SRR was reduced to 100% for most of the tests. A maximum entrainment speed of 10 m/s (sliding speed of 10 m/s for SRR = 100%, and 9 m/s for SRR = 180%) was tested on the EHD. Each test was done at fixed speed, pressure and SRR. The contact and test conditions in the EHD machine are shown in Table 5 and Table 6.

A fresh steel ball was used for each test and a new, non-rubbed track radius was chosen on the disc. The EHD test specimens and EHD test chamber were cleaned similarly to the ETM procedure described above. The pot was filled completely with the soap powder.

Table 5: Test conditions employed in the high-speed EHD rig for soap film observation tests

Maximum Hertzian pressure [GPa]	0.53, 0.98
Test temperature [°C]	25 (room temperature)
SRR	-100%, -180%
Entrainment speed (u_e) [m/s]	Up to 10

Table 6: Contact size of EHD ball-disc contact with different disc materials

Disc material	$P_{\max}$ [GPa]	P_{mean} [GPa]	a [μm]	W [N]
Glass	0.42 / 0.53	0.28 / 0.36	106.1 / 133.7	10 / 20
Sapphire	0.98	0.66	98.5	20

3.4 Post-Test Analysis of the Specimens

Further techniques were used to get deeper insights into the nature of the lubricating soap films that may have been deposited on the test specimens. The topography of the specimens' surfaces and of any soap deposited films was characterised. The chemistry of any deposited films in the rubbing track was also studied.

3.4.1 Surface Characterisation

The surfaces of the specimens were characterised using tactile and optical measurement systems prior and post-tests. In order to analyse roughness, a tactile profilometer Taylsurf from Taylor Hobson was utilised. This machine uses a stylus which moves along the surface and detects topographic defects.

For this work, two roughness parameters, namely the arithmetic mean roughness, Ra , and the root mean square roughness, Rq , are taken into consideration. Ra is the arithmetic mean of the profile along the centre line, see equation (3.2),

$$Ra = \frac{1}{L} \int_0^L |z| dx \quad (3.2)$$

where $z(x)$ is the height above the centre line of the surface and L is the length of the profile (12). The Rq value is the standard deviation of the surface height from the centre line (12), see equation (3.3).

$$Rq = \sqrt{\frac{1}{L} \int_0^L z^2 dx} \quad (3.3)$$

To investigate the topography of the specimens in more detail and to get simultaneous images of the surfaces, an optical confocal microscope Olympus OLS5000 LEXT was also employed. This optical system uses a spatial pinhole which only allows focused laser light to reach the detector. The laser then scans every point in a given area and is thereby able to outline a 3D representation of the surface by measuring at different depths. This also allowed to assess any surface features, as well as measure the thickness of any deposited films on the specimens following the procedure described in 3.2.3.

3.4.2 Chemical Analysis of Lubricating Layer

To analyse the chemistry of the lubricating layers Raman spectroscopy with a 532 nm laser was used. Several spectra were stitched together with an integration time of 1s for 100 accumulations and a range from -200 to 3500 cm^{-1} . The obtained spectra can be used to identify significant peaks which relate to molecule bonds and therefore give implications of any chemical interactions that could have occurred.

To further study chemical components of any lubricating layers, scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) was also employed. Images of the rubbing track (field of view of $500\text{ }\mu\text{m}$) were taken with a loading energy of 20 keV and a beam current of 3 nA (41).

3.5 Thermoanalytical Testing

The results of this study, which will be presented later, indicated that contact temperatures may play an important role in soap lubrication mechanisms. To help to better understand the effect of temperature on soap properties Differential Scanning Calorimetry (DSC) was used to characterise the effect of temperature on the fresh soap powders. A low temperature DSC was used first where the temperature was increased in controlled manner to 400°C .

To confirm the results and detect further changes with increased temperature, another DSC-TGA was used to simultaneously output the thermogravimetric analysis as well as differential scanning calorimetry data. The analysis was done under argon atmosphere and the temperature was increased from 20°C to 800°C with a heating rate of 20 K/min . This allowed to identify possible melting temperatures, transition temperatures as well as mass changes and temperature stabilities.

3.6 Dry Wire Drawing Soaps

Typical wire drawing powdered soaps were tested: two calcium stearates and a sodium stearate. These soaps were fully formulated as provided by the sponsor's (Bekaert) supplier. The material properties and the grain size distribution of the soap powders are listed in Table 7 and Table 8 respectively. The soaps were chosen to cover a wide range of different total fatty acid content as well as different soap metals.

Table 7: Basic chemical composition of tested soap powders (from Bekaert internal specifications)

Ca-Soap 1	Ca-Soap 2	Na-Soap
Calcium stearate C18	Calcium stearate C18	Sodium stearate C18
Free of sodium nitrite and barium stearate	Contains 3% sodium soap; free of TiO ₂ , nitrite, borax	Addition of mineral salts, borax and Na ₂ SiO ₃
35 ± 1.5 % total fatty acids	55 % total fatty acids	78 % total fatty acids
Insoluble in water	Insoluble in water	Water soluble

Table 8: Grain size distribution of tested wire drawing soaps (from Bekaert internal specifications)

Grain Size / mm	Distribution of Soap / %		
	Ca-Soap 1	Ca-Soap 2	Na-Soap
≥ 2.000	15 – 40	< 2	12 – 28
< 2.000 ≥ 1.000	25 – 40	2 – 25	30 – 55
< 1.000 ≥ 0.500	12 – 30	15 – 35	10 – 30
< 0.500 ≥ 0.250	2 – 15	10 – 35	5 – 20
< 0.250 ≥ 0.125	2 – 10	5 – 20	3 – 11
< 0.125	2 – 10	0 – 25	0 – 10

Since the fully formulated soaps have very different fatty acid content, some pure calcium (calcium dioctadecanoate) and sodium (sodium octadecanoate) stearates were also tested to assess potential effects of fatty acid content on friction performance.

3.7 Borax Coating

In the real dry wire drawing application, the wire is drawn through a powdered soap box, and usually a lubricant carrier, applied as a wire coating, is used to aid the soap to adhere to the wire as it passes through the soap box (68). This carrier forms a tough wire coating that increases the roughness and therefore, facilitates the soap pick-up onto the wire. One commonly used lubricant carrier is Neobor borax (68) which is a disodium tetraborate pentahydrate. For this thesis, the commonly used term ‘borax’

will be used to refer to this coating.

Most of the ETM tests performed below are done without a borax coating. However, some discs were coated prior to the tests to study the effect of it on friction and to mimic real wire drawing material-pairs. The tests with borax coated discs are indicated appropriately.

In these specified experiments, the ETM discs were coated with borax prior to testing similar to the process for steel wires. For this, a bath with an 80g/l borax-water solution at a temperature of 85°C was prepared. The ETM discs were kept in this bath for about 5 min, so the discs reach the same temperature as the bath, after which the discs are dried in a furnace at 130°C for 20 min.

3.8 Universal Wire Drawing Test Machine (UWT)

Parallel to the bench-tests in the lab, wire drawing tests were performed in a universal wire drawing test machine (UWT) at Bekaert site. The present author helped to interpret the results, but it should be noted that these actual tests were conducted by the personnel employed by the industrial sponsor (Bekaert) of this PhD project. The results were used to compare the trends achieved in the lab tests and those in the real application and hence, to affirm the validity of the lab tests employed in this study in terms of them producing results applicable to real wire drawing.

The UWT is a single-pass drawing machine. A highly simplified illustration is shown in Figure 3-4. The wire is passed from a capstan through the soap box to the drawing die. The drawn wire is then recoiled onto another capstan. Several sensors are attached to measure and control the backpull force and drawing force, as well as the inlet and outlet velocity of the wire. The drawing speeds tested ranged from 5 m/min (0.08 m/s) to 250 m/min (4.17 m/s) which covers the speed used in the lab test (sliding speeds up to 3.6 m/s). The backpull forces were controlled to be 100 N. The final drawn wire diameter was 1.50 mm with a reduction ratio of 25%. The wire was pre-coated with borax and the same sodium soap from the ETM tests as described above was used in the UWT tests.

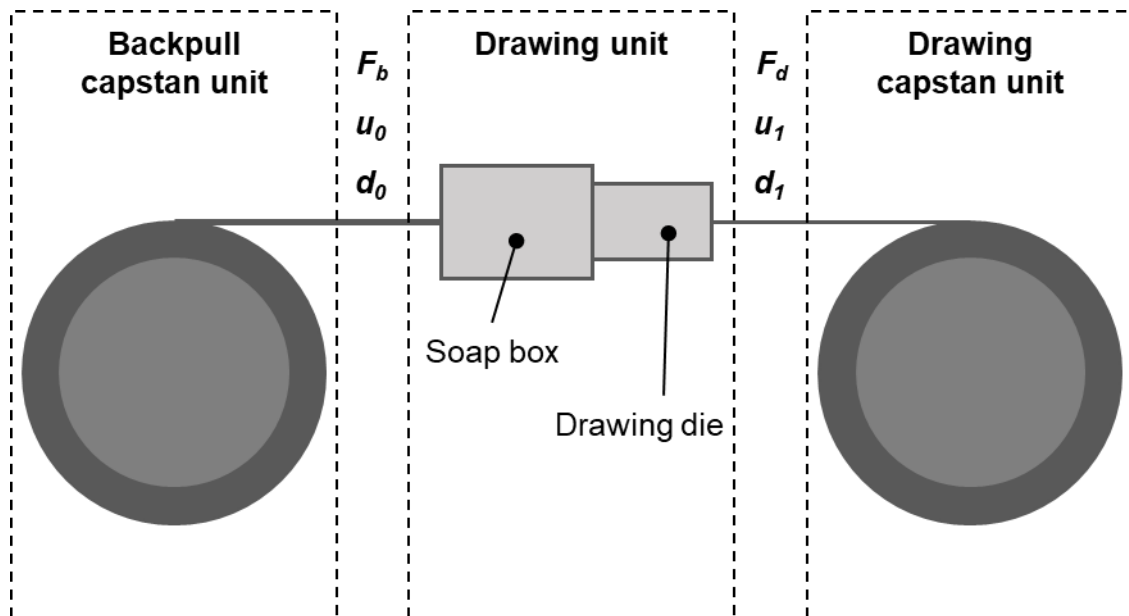


Figure 3-4: Simplified schematic of the universal wire drawing test machine (UWT)

4 Friction Behaviour of Dry Wire Drawing Soaps

This chapter describes the frictional behaviour of dry wire drawing soaps as measured in the ETM experiments. The influence of different parameters on friction is analysed and discussed. Large parts of the contents of this chapter have already been published by the present author in the open access paper (41).

4.1 Effect of SRR

The influence of slide-roll-ratio (SRR) on soap friction was investigated by performing a series of tests at 3.0 GPa pressure and 1.5 m/s entrainment speed over the range of SRR from 0% (pure rolling) to 180% (close to pure sliding) for both the Ca and Na based commercial soaps described earlier. The results of these tests are shown in Figure 4-1. Three repeats were done for each test. The solid line plots are averaged data over three repeats for each soap and the error bars show the standard deviations from these three repeat tests.

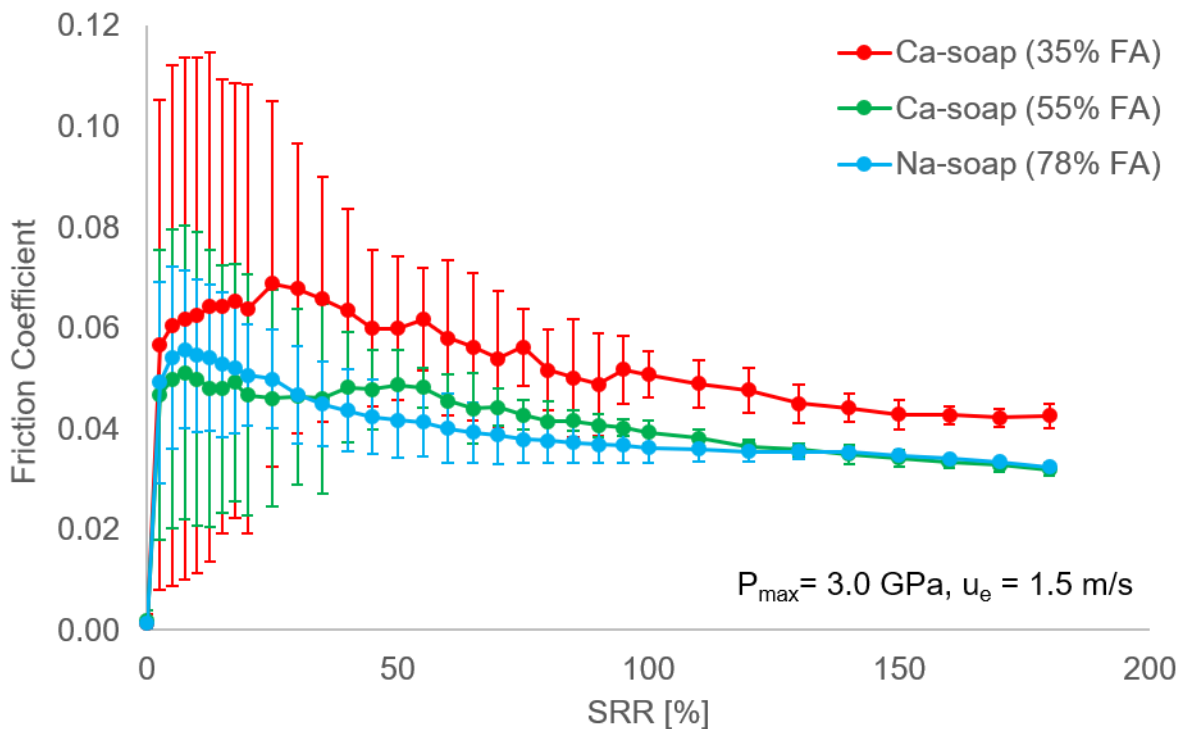


Figure 4-1: Measured ETM friction coefficient for sodium and calcium soaps over a range of SRR (i.e. traction curves) from 0% (pure rolling) to 180% (close to pure sliding).

It can be seen that for each soap, friction is very close to zero in pure rolling as may be expected and then raises rapidly with increasing SRR, reaching a peak relatively quickly between about 5% and 15% SRR. As SRR increases further friction decreases. The rate of decrease slows with increase of SRR so that friction reaches a plateau at about 150% SRR for all soaps tested. This suggests that friction at 180% and pure sliding (200%) is similar, which further supports the use of 180% as the SRR in other tests in this work.

Figure 4-1 also shows significant variability in results between repeat tests at low SRRs but good repeatability at high SRRs of about 100% and over. The variability at low SRR is irrelevant in practical terms as these conditions are far removed from actual wire-die contact conditions (pure sliding). All further tests below were conducted at 180% SRR. However, the variability at low SRR and the overall trend of friction with SRR may provide useful information in terms of mechanisms of soap lubrication. For example, they may indicate that soap lubrication is sensitive to shear rates applied or to contact flash temperatures which increase with increasing SRR. The mechanisms of soap lubrication will be discussed later in this thesis.

A notable observation in these tests is that the coefficient of friction (COF) at high SRR is as low as 0.03 to 0.04 for all soap formulations tested here. These values are comparable to friction coefficients in equivalent contacts lubricated with oils under full film conditions. Therefore, notwithstanding the relative complexity in their frictional behaviour, it is clear that powder soaps used in wire drawing are very effective in reducing friction coefficients in high-sliding, high-pressure contacts.

4.2 Effect of Speed

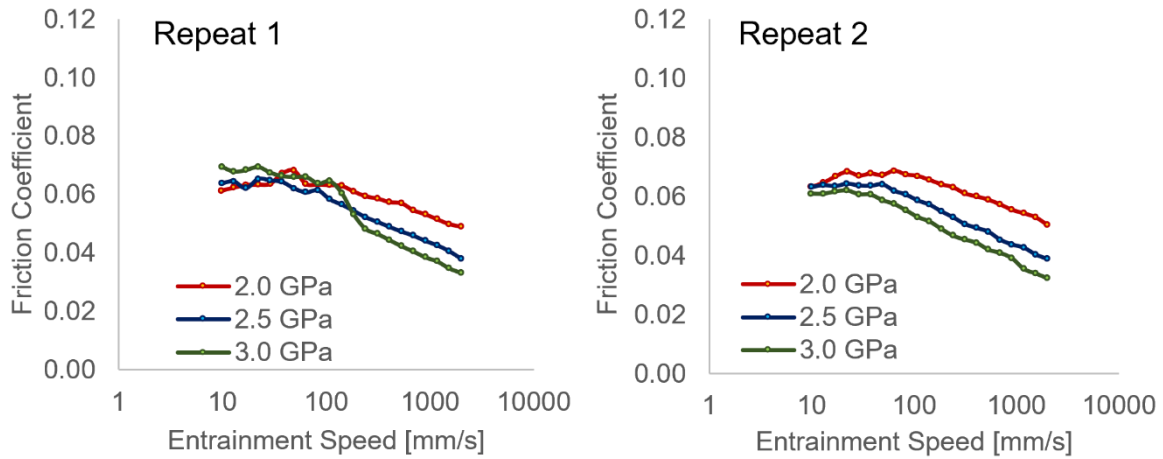
The effect of entrainment speed on friction is shown in Figure 4-2 at three different Hertzian maximum pressures. There are two repeat tests for each soap and the tests show good repeatability. In these tests the SRR was kept constant at 180%, so increasing the entrainment speed means that sliding speed is also increased. For the sodium soap, the COF is relatively constant at 0.06 - 0.07 at low speeds but then decreases with increasing entrainment speed past a speed of around 100 mm/s (Figure

4-2a). At highest speeds and pressure tested (2.0 m/s, 3 GPa), the COF is as low as 0.033; it does not appear to have reached a plateau as the gradient of friction curves is still negative, so it is likely that friction would be even lower at higher speeds.

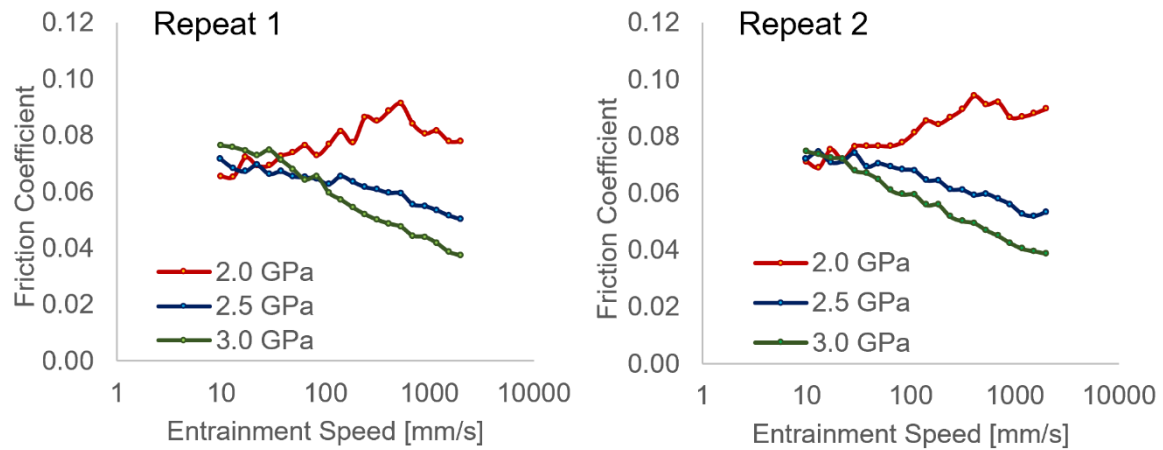
For the calcium soap with low FA content of 35%, a similar trend is seen for higher pressures of 2.5 GPa and 3.0 GPa (Figure 4-2b), with a COF of 0.07 at low speed and falling to below 0.04 at the highest speed and pressure. There is less of a plateau below 100 mm/s, instead friction seems to be decreasing with increasing speed relatively monotonically. A different trend, however, is seen for a pressure of 2.0 GPa, where friction increases with increasing speed. To confirm this anomaly the result of a repeat test is also shown, and it exhibits an almost identical behaviour. The reasons for the different frictional behaviour of this calcium soap (35% FA) at lower pressure is not clear at this stage but some possibilities are discussed later.

Highest friction, with COF ranging between 0.07 and 0.10 at low speeds, was measured with the calcium soap with medium FA content of 55% (Figure 4-2c). There is a plateau up to 100 mm/s, after which friction decreases with increasing speed, similar to the high FA content sodium soap. At the highest pressure and highest speed (3.0 GPa, 2.0 m/s) friction is the lowest at around 0.030, where it seems to have not yet reached a plateau, again, very similar to the sodium soap. A further decrease in friction could therefore also be likely here. However, for the 2.0 GPa, friction slightly increases at higher speeds from 1.5 m/s onwards, more similar to the behaviour of the low FA content calcium soap at a pressure of 2.0 GPa.

a) Na-soap (78% FA)



b) Ca-soap (35% FA)



c) Ca-soap (55% FA)

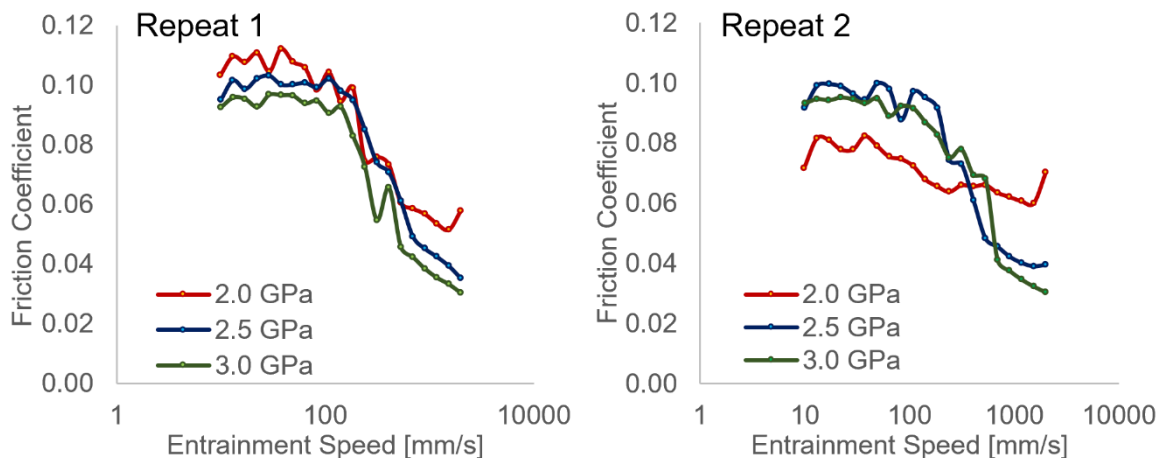


Figure 4-2: Measured ETM friction coefficient over a range of entrainment speeds from 10 to 2000 mm/s (i.e. 'Stribeck' type curves) at $SRR = 180\%$ and pressures (2.0, 2.5, 3.0 GPa); a) sodium soap (78% FA), b) calcium soap (35% FA), and c) calcium soap (55% FA).

4.3 Long-term Friction Evolution of Soaps

Above tests showed that both sodium and calcium soap powders consistently achieved low COFs particularly at high sliding speeds and pressure. However, each of the tests above (sweeps through SRR or speed) are relatively short in duration. The obvious question is whether this low friction behaviour persists over a longer duration at any given combination of speed, SRR and pressure.

To investigate this, ETM tests were conducted over a period of one hour at fixed contact conditions, SRR of 180%, entrainment speed of 1.8 m/s, and pressures of 2.0 GPa, 2.5 GPa or 3.0 GPa. The results of these tests are shown in Figure 4-3. The tests with the sodium soap show that there is a short running-in period in the beginning where friction decreases to low and stable values. In general, friction is very low with the sodium soap ranging between 0.03 and 0.04. At 3.0 GPa, some variation in friction is seen towards the end of the test.

For the calcium soap with low fatty acid content of 35%, a similar running-in phase is seen where friction decreases in the beginning of the test, e.g. for 2.0 GPa the COF decreases from about 0.09 to about 0.04 over the first two minutes. In comparison to the sodium soap above, the friction here shows some more fluctuations rather than smooth curves, especially towards the end of the test.

Comparable trends are also seen with the medium FA content calcium soap (55%) where friction decreases in the beginning from about 0.04 – 0.06 to about 0.03 – 0.04, and again, more instabilities are seen after some time.

In general, for all three cases the running-in period is very short after which low, stable friction was produced over a long period ranging between 0.03 and 0.04. This indicates that whatever the nature of the lubricating film formed by the soaps, they are persistent and stable, and the soaps are being continuously entrained into the contact in these ETM tests. However, some instabilities are seen near the end of the 1-hour tests, perhaps indicative of occasional starvation. During the real wire drawing process, the wire only spends a fraction of a second in contact with a die, but it goes through a number of dies to reach the final reduction.

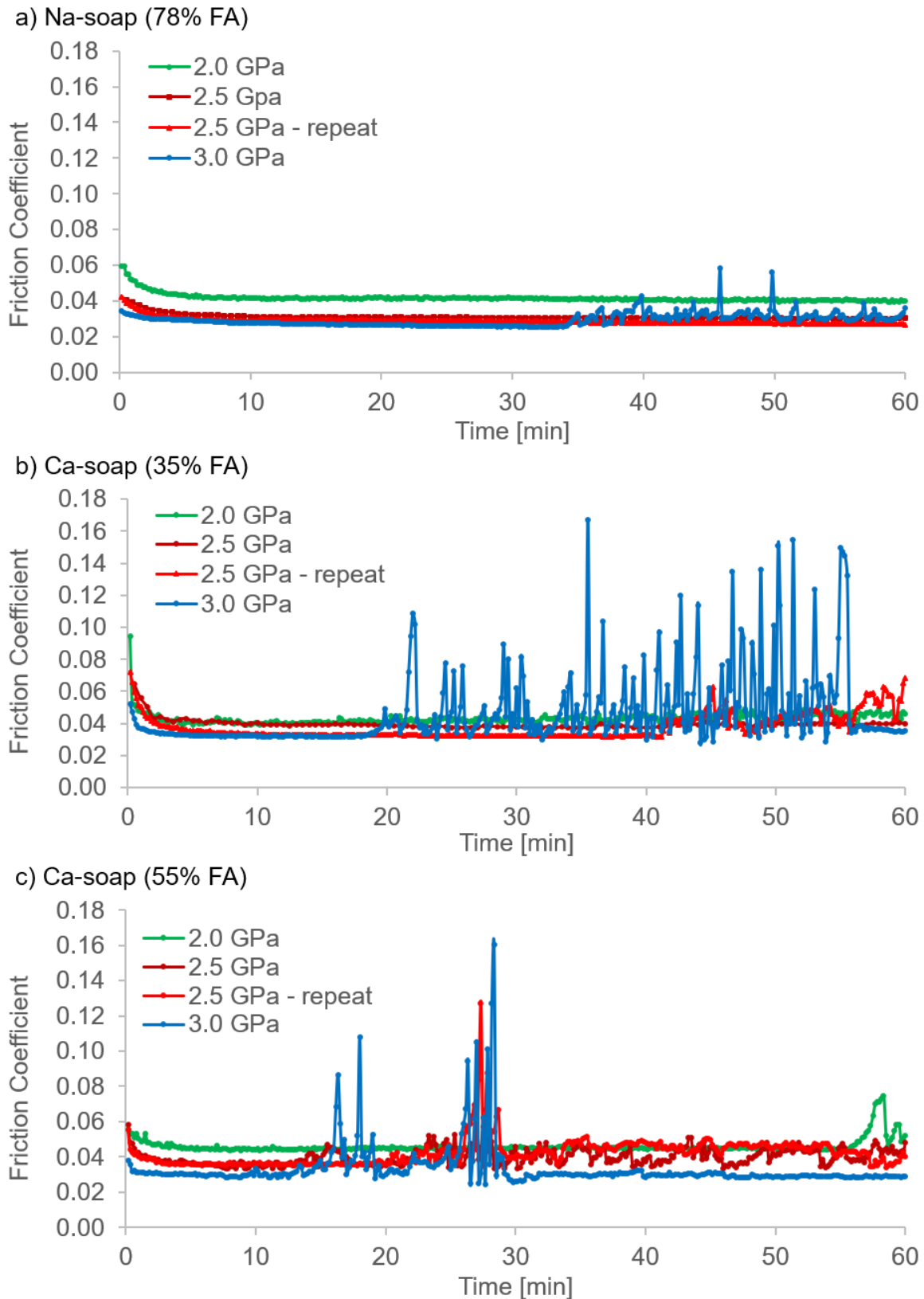


Figure 4-3: Measured ETM friction with calcium and sodium soaps over a 1-hour test period at $SRR = 180\%$, $u_e = 1.8$ m/s, $T = 40^\circ\text{C}$.

4.4 Effect of Contact Pressure

Pressure Effect during Stribeck Test

Figure 4-2 also shows the influence of contact pressure on friction. At lower speeds for the sodium soap (Figure 4-2a), only small differences in the COF are seen with all three Hertz pressures tested (2.0, 2.5, 3.0 GPa). At high speeds, friction decreases with increasing pressure and friction is always lowest at the highest pressure of 3.0 GPa.

For the calcium soap with 35% FA content, similar effects can be seen. At lower speeds, pressure has only little effect on friction. From speeds of about 50 mm/s and higher, friction drops with increasing pressure. Again, the highest pressure leads to the lowest friction at high speeds.

The calcium soap with a FA content of 55%, shows a slightly worse repeatability at low speeds and lowest pressure tested (2.0 GPa), where friction varies significantly. For 2.5 GPa and 3.0 GPa pressures, the difference in friction remains very low throughout the test range, yet again, an increase in pressure leads to a decrease in friction.

The test results for the Stribeck tests (Figure 4-2) were replotted in Figure 4-4 to show the maximum shear stress in the contact instead of the friction coefficient. The maximum shear stress was calculated by multiplying measured COF and calculated maximum Hertzian pressure for a given speed. Using this, it becomes clear that with increasing speed, shear stress decreases for the sodium soap (Figure 4-4a). At speeds above 400 mm/s, the shear stress is similar for all pressures tested with lowest maximum shear stress of around 100 MPa.

Same trends were seen for the calcium soap with low fatty acid content at the higher pressures of 2.5 and 3.0 GPa where shear stress seems to decrease with increasing speed (Figure 4-4b). For these two pressures, the shear stress curves overlap at speeds of around 200 mm/s after which there are no noticeable differences. The lowest shear stress reached is around 115 MPa. Interestingly, the 2.0 GPa curve also overlaps at a speed of around 200 mm/s but increases further with increasing speed, and only starts decreasing at around 500 mm/s. Again, the reason for this behaviour is unclear, but some possible reasons are discussed later on.

For the calcium soap with medium fatty acid content (Figure 4-4c), the shear stress also increases with increasing pressure at low speeds. With increasing speed, shear stress decreases and the curves for 2.5 and 3.0 GPa are overlapping at speeds above 400 mm/s and they reach a minimum of around 90 MPa at highest speeds tested. In the first repeat test, the 2.0 GPa trends seem to follow the other pressures but in the second repeat, the shear stress seems to stay at a level of around 120 MPa with a slight upwards trend. This is similar to the trends of the calcium soap with 35% fatty acid content at a pressure of 2.0 GPa (Figure 4-4b).

Figure 4-5 plots the maximum shear stress vs. maximum Hertzian pressures tested. At lower speeds, all three soaps exhibit a positive linear relationship between shear stress and pressure. For the sodium soap (Figure 4-5a), at speeds above 0.5 m/s, shear stress seems to become independent of pressure as was described above. Interestingly, for the low FA (35%) calcium soap (Figure 4-5b), the shear stress vs. pressure relationship changes and a downward trend is observed at higher speeds above 0.3 m/s. This drop in shear stress with increasing pressure could perhaps indicate that the soap shows easier plastic flow at higher pressures. For the medium FA (55%) calcium soap (Figure 4-5c) a more 'erratic' behaviour is recorded which seems to be a mixture between the trends seen for the high FA (78%) sodium soap and the low FA (35%) calcium soap. It is clear that the combination of speed and pressure matters for the low friction behaviour of the soaps. The observed trends shown in Figure 4-4 and Figure 4-5 are important in terms of explaining the nature of the soap films and will be discussed later.

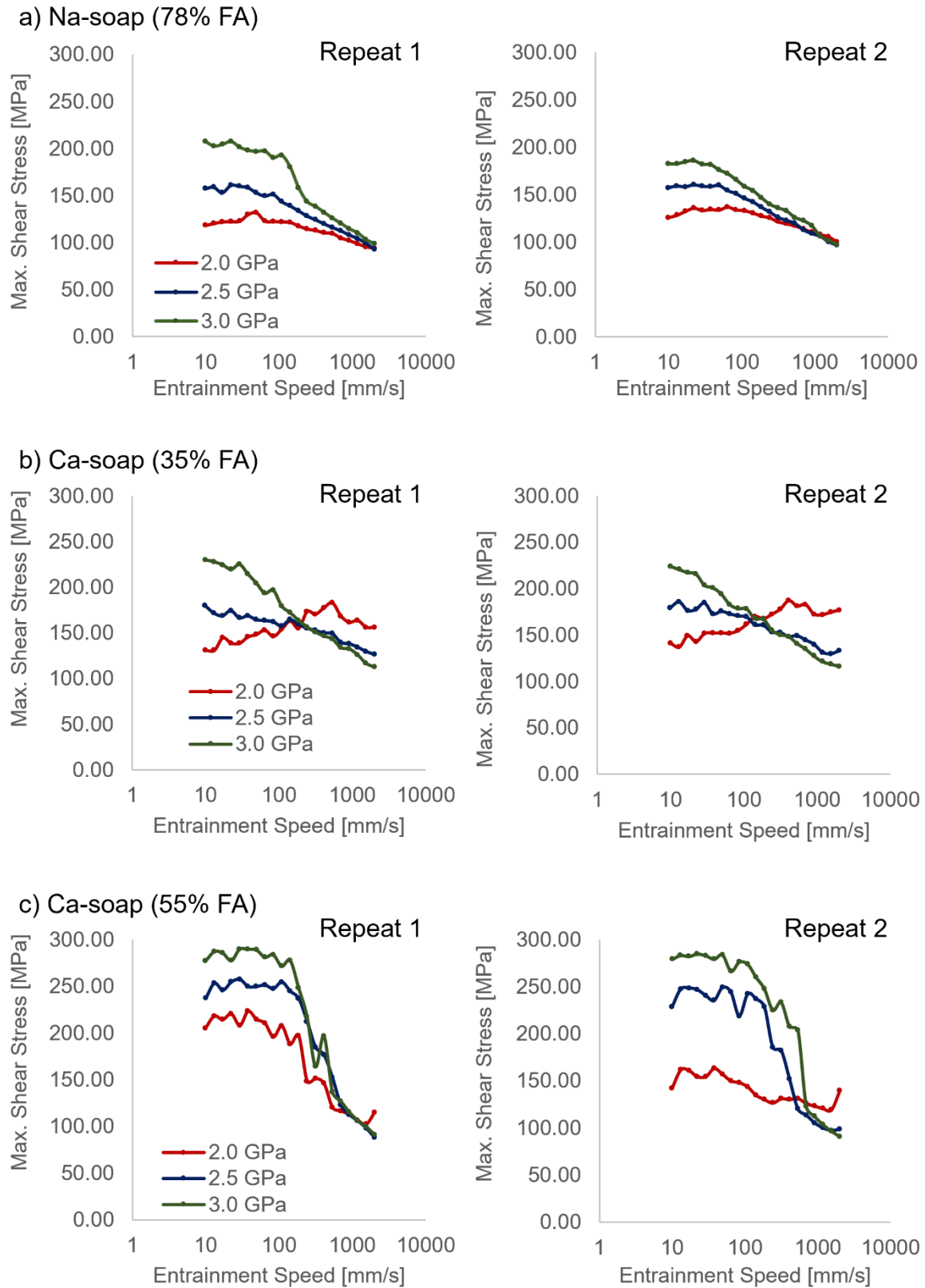


Figure 4-4: Maximum contact shear stress over a range of entrainment speeds and pressures (2.0 , 2.5, 3.0 GPa) at SRR = 180% (replotted from tests in Figure 4-2); a) sodium soap (78% FA), b) calcium soap (35% FA), and c) calcium soap (55% FA).

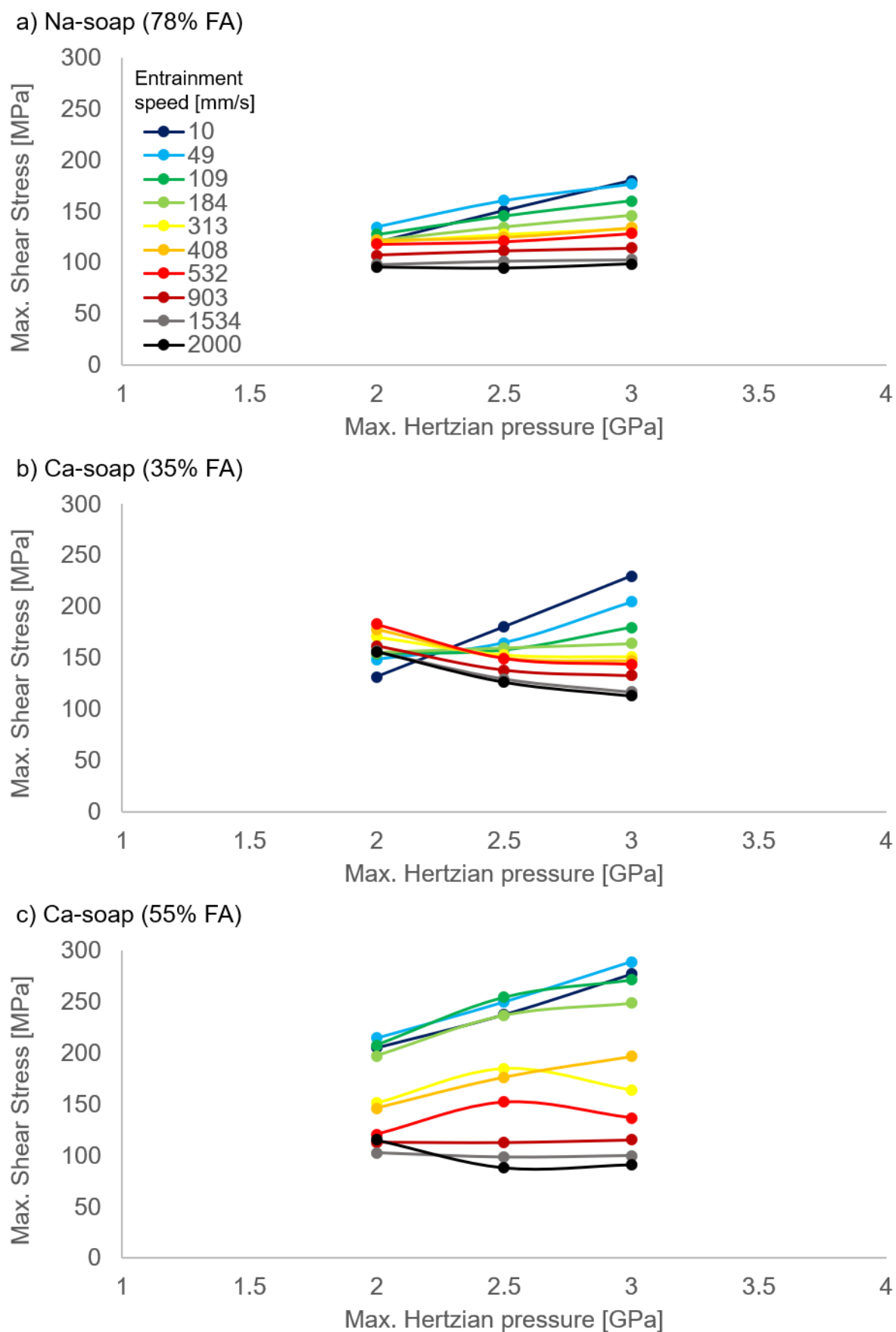


Figure 4-5: Max. shear stress vs. max. Hertzian pressure over range of speeds tested, replotted from Figure 4-4.

Pressure Effect during Long-Term Friction Test

The effect of pressure in a long-term test at fixed conditions is demonstrated in Figure 4-3. Here, the SRR is kept constant at 180% and the tests are done at pressures of 2.0 GPa, 2.5 GPa or 3.0 GPa. Each curve represents one test. For all three soaps, it is obvious that an increase in pressure leads to a decrease in friction. The sodium soap (Figure 4-3) shows very good repeatability and very stable friction with slight instabilities developing at 3.0 GPa after about 35 min. Stable COFs with the sodium soap are as low as 0.042, 0.032, 0.029 at 2.0 GPa, 2.5 GPa and 3.0 GPa respectively. Friction increases up to 0.060 at 3.0 GPa during the instabilities.

Both calcium soaps (Figure 4-3b, c) show stable COFs in the beginning of the tests but friction begins to vary around 15-20 min, more or less for all pressures. For the highest pressure, friction can get as high as 0.160, indicating occasional starvation or mixed lubrication. Stable COFs with the low FA content calcium soap (35%) are between 0.033 and 0.040. For the calcium soap with the medium FA content of 55%, stable friction coefficients of 0.044, 0.036 and 0.029 are achieved for 2.0 GPa, 2.5 GPa and 3.0 GPa respectively.

4.5 Influence of Soap Formulation

Influence of Soap Type

The soap type has a major effect on friction. In all friction tests, out of three soaps tested, the sodium soap with high fatty acid content (78%) consistently achieves lowest COFs, closely followed by calcium soap with 55% FA while the calcium soap with low fatty acid content (35%) had significantly higher friction. The lowest COF measured at high SRR, high pressures and high speeds is around 0.03 for the sodium soap (e.g. Figure 4-2 and Figure 4-3). For the calcium soap with low FA content (35%) the lowest COF was measured to be around 0.04. Reasons for this trend are discussed later on, but to better understand this, the soap films have to be analysed further.

The friction behaviour of the calcium soap with medium fatty acid content of 55% seems to be a mixture between the behaviour of the sodium soap (78% FA content) and the low FA content calcium soap (35%). In absolute values, the achieved friction

values are very low and very similar to the sodium soap with the lowest COF at high SRR, high speed and high pressure to be around 0.03 (e.g. Figure 4-2c). These trends suggest that the amount of fatty acid content could perhaps be the reason for the low friction produced with the soaps rather than the metal component to which the soap is bound.

Influence of Fatty Acid Content

To study the effect of the fatty acid content on friction, a series of tests was performed with pure powdered calcium and sodium stearates, simulating a fatty acid content of ~100%. The measured ETM friction with varying entrainment speeds (e.g. Stribeck curve) at fixed SRR of 180% and pressures is displayed in Figure 4-6.

The results in Figure 4-6 show that friction decreases with increasing speed and increasing pressure as was also observed with all formulated soaps above. Moreover, for the sodium stearate, a short friction plateau is seen at low speeds up until 100 mm/s, whereas for the calcium stearate, friction seems to decrease almost immediately with increasing speed. In any case, more fluctuations are seen with the calcium stearates, yet in absolute values the measured friction coefficients seem to be very similar between the calcium and sodium stearates. This is in contrast to the tests shown above with formulated soaps where the sodium soap showed consistently lower friction than the calcium soap, but there, the sodium soap had a much higher fatty acid content, too. In combination, these trends suggests that the amount of fatty acid content has a strong, if not the dominant, effect on friction rather than that the metal content being the only factor in determining the friction behaviour of soaps. However, the effect of other additives included in the formulated soaps have not been tested and hence, a clear conclusion cannot be drawn.

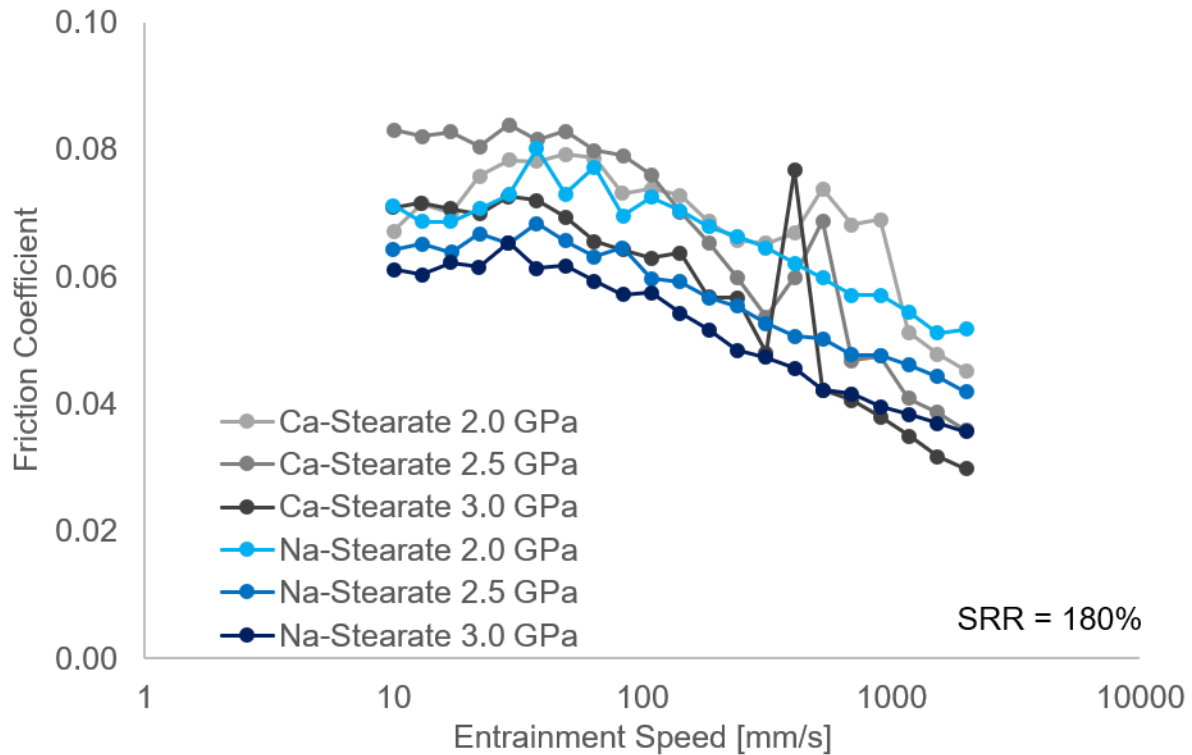


Figure 4-6: ETM friction measured at varying speeds (Stribeck curves) with pure sodium and calcium stearates.

The repeat tests for Figure 4-6 are shown in Figure 4-7. Good agreement between repeats is given for the calcium stearate at high speeds; only some differences are seen at lower speeds. For the sodium stearate good repeatability is given throughout the tests for high pressures of 2.5 GPa and 3.0 GPa; minimal fluctuations are seen at lower speeds at lowest pressure of 2.0 GPa.

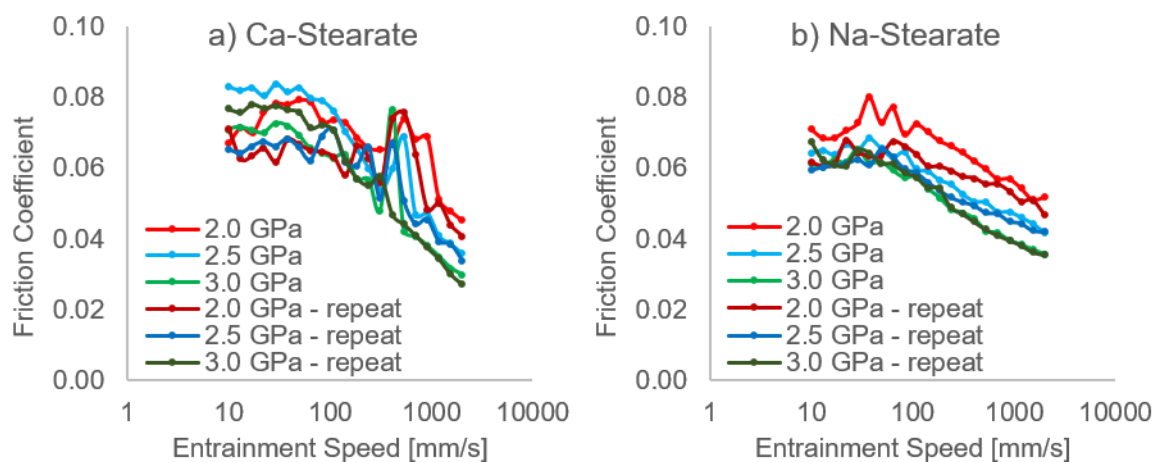
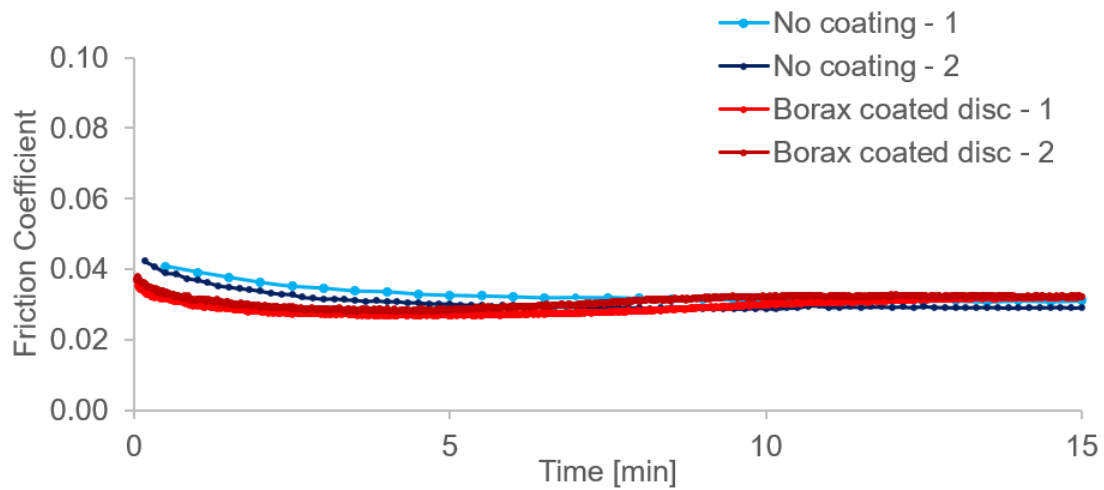


Figure 4-7: ETM Stribeck tests with repeats for a) calcium and b) sodium stearates.

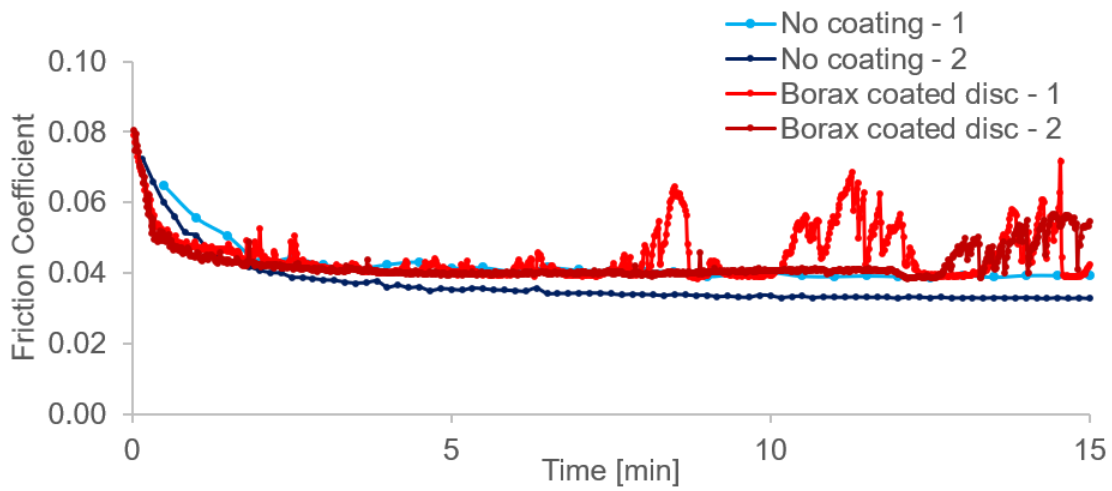
4.6 Influence of Borax coating

In wire drawing application, the wire is usually coated with a borax coating (disodium tetraborate pentahydrate) prior to drawing to increase roughness and, thereby promote soap adhesion onto the wire. To mimic this coating and identify the effect of it on friction in the ETM test set-up, some discs were coated with borax as described in chapter 3.7. The effect of the borax coating compared to the standard tests above without any coating on the discs is shown in Figure 4-8. A long-term test at fixed conditions (2.5 GPa, 180% SRR, and entrainment speed of 1.8 m/s) was chosen rather than a 'short' Stribeck or Traction curve test to reduce the variability of parameters and therefore, to see a direct influence of the coating. Moreover, the greater duration of the test also allows to test the durability of the coating, e.g. the identification of anomalies could indicate breakage of the coating.

a) Na-soap (78% FA)



b) Ca-soap (35% FA)



c) Ca-soap (55% FA)

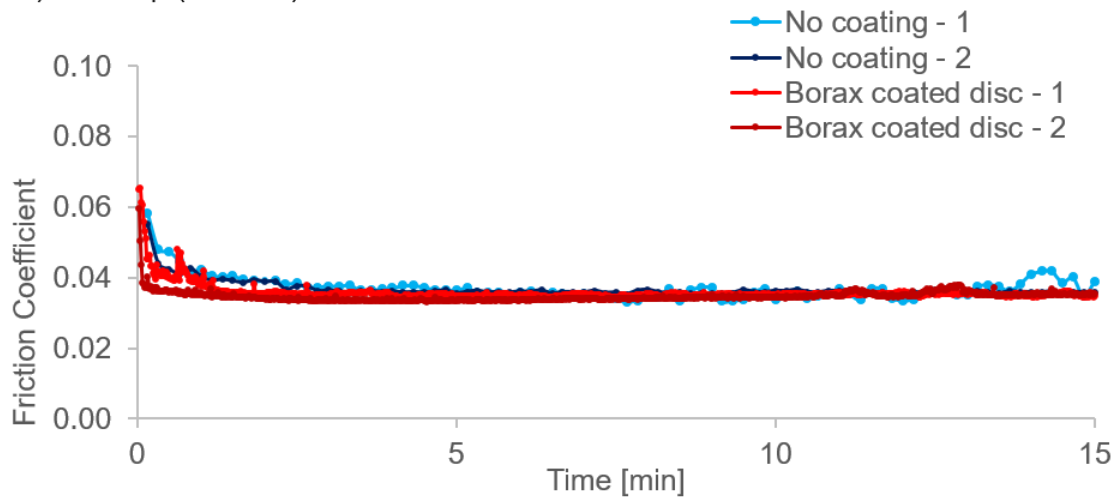


Figure 4-8: Effect of borax coated discs on friction performance in ETM ($u_e = 1.8$ m/s, $SRR = 180$ %, $P_{max} = 2.5$ GPa).

In all three cases the results show that the running-in period is shorter and friction is lower during running-in when the borax coated discs were used compared to standard non-coated discs. For the sodium soap (Figure 4-8a), the borax coating on the discs resulted in minimal lower friction in the initial phase of the test, but friction behaviour becomes identical to the tests with non-coated discs after around 5 minutes. This might indicate, that at the start of the test, the borax coating improved soap pick-up in addition to the ETM scoop but after a few minutes the coating might have been removed. The lowest COF achieved with the borax coated discs is around 0.029 which is more or less the same as that achieved with no coating.

In case of the medium FA content (55%) calcium soap (Figure 4-8c), no considerable difference in friction between the standard discs and the coated discs can be seen, except for in the running-in period up until ~3 min of the test. For the low FA content calcium soap (Figure 4-8b), substantial variations in friction are seen when the borax coated discs were used.

Overall, it seems that the borax coating might increase soap pick-up and therefore, further decreases friction during a test with short duration. However, the difference in friction is very small and thus, can be considered negligible in the ETM friction tests. The scoop set-up in the ETM is capable of sufficiently providing soap to the contact similarly to the borax coating on the discs. In addition, the persistency and homogeneity of the coating might change the conditions during the test and lead to an additional parameter that needs to be controlled. The difficulty in predicting and distinguishing the coating behaviour, as well as the negligible discrepancy in friction, justify the use of standard steel discs without any coating throughout the thesis.

4.7 Influence of Soap Grain Size Distribution

The size of the soap particles is another factor that could affect the formation of a lubricating layer (55) and therefore, influence the frictional behaviour. To study this, soap batches with different size distributions were prepared and their frictional behaviour studied.

All above mentioned tests were done with the “as supplied” batch which has the

distribution specified for each soap in Table 8 (chapter 3.6). This is a mixture ranging from particle size below 0.125 mm to above 2.0 mm. To study the effect of different grain sizes on friction, the industrial partners were asked to sieve the sodium soap and the low FA content calcium soap (35%) to the fractions in Table 9.

Table 9: Soap samples with different grain size distributions

Legend name	Grain Size / mm	Distribution
As supplied	$< 0.125 - \geq 2.000$	See Table 8 (chapter 3.6)
0.125 – 0.5 mm	0.125 – 0.500	100 %
1.0 – 2.0 mm	1.000 – 2.000	100 %

The effect of different soap particle fractions of the sodium soap is shown in Figure 4-9. The main observation here is that for all pressures, the grain size of the sodium soap particles does not affect friction at very high SRRs and sliding speeds.

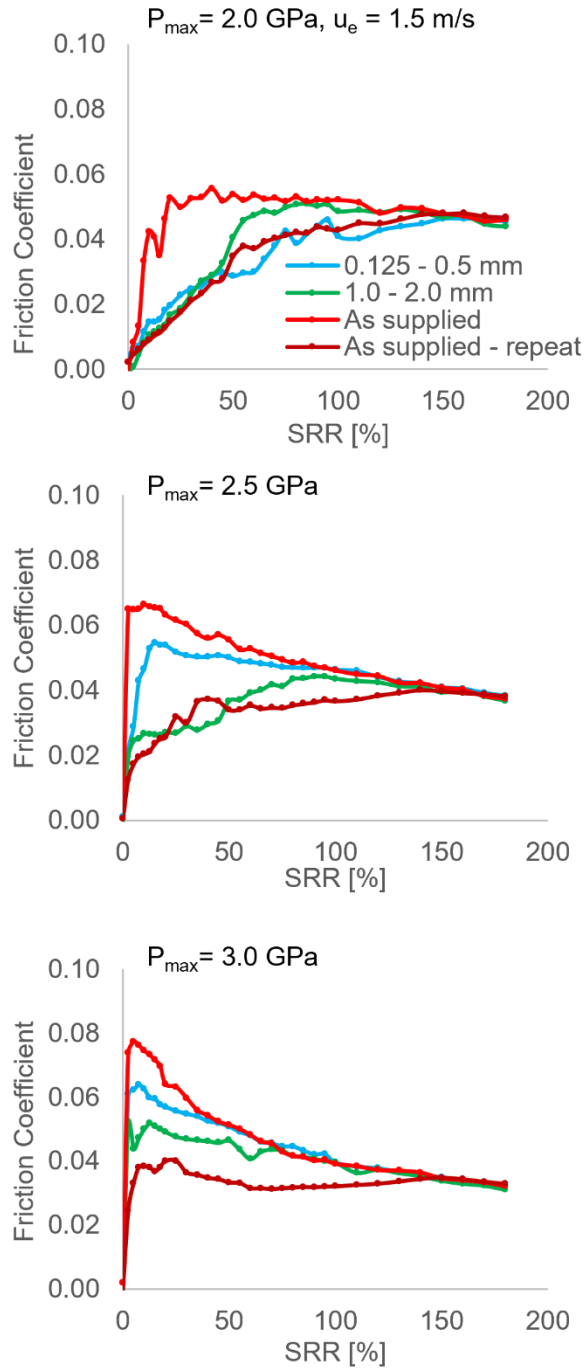
The traction curves (Figure 4-9a) show some disparities between soap distributions at lower SRR below 150%. As described above (section 4.1), repeatability at low SRR is very poor and only at high SRR above 100% does it improve. Therefore, only high SRRs close to pure sliding, and closer to the real application, are considered. In this case, it becomes clear that at all pressures, the different grain sizes lead to identical friction behaviour as the “as supplied” fraction.

Considering the ‘Stribeck’ curves with the sodium soap (Figure 4-9), it can be seen that again, at all pressures, friction is almost identical between the “as supplied” and the “1.0 – 2.0 mm” fractions throughout the speed range. Contrary to the assumption of full film formation with smaller grain sizes (55), the smallest fraction of “0.125 – 0.5 mm” lead to slightly higher friction at low speeds with a COF of around 0.08 at a speed of 10 mm/s for all pressures. This fraction also shows more fluctuations throughout the tests. At high speeds above 500 mm/s, the small soap fraction shows very similar COFs as the other fractions. In all cases, however, friction decreases with increasing speed and reaches a very low COF of 0.033 at 3.0 GPa and 2.0 m/s.

In general, it seems that the effect of grain size distribution on friction is very negligible for the sodium soap, especially considering that wire drawing involves very high

speeds.

a) Traction Curves with Na-soap



b) 'Stribeck' Curves with Na-soap

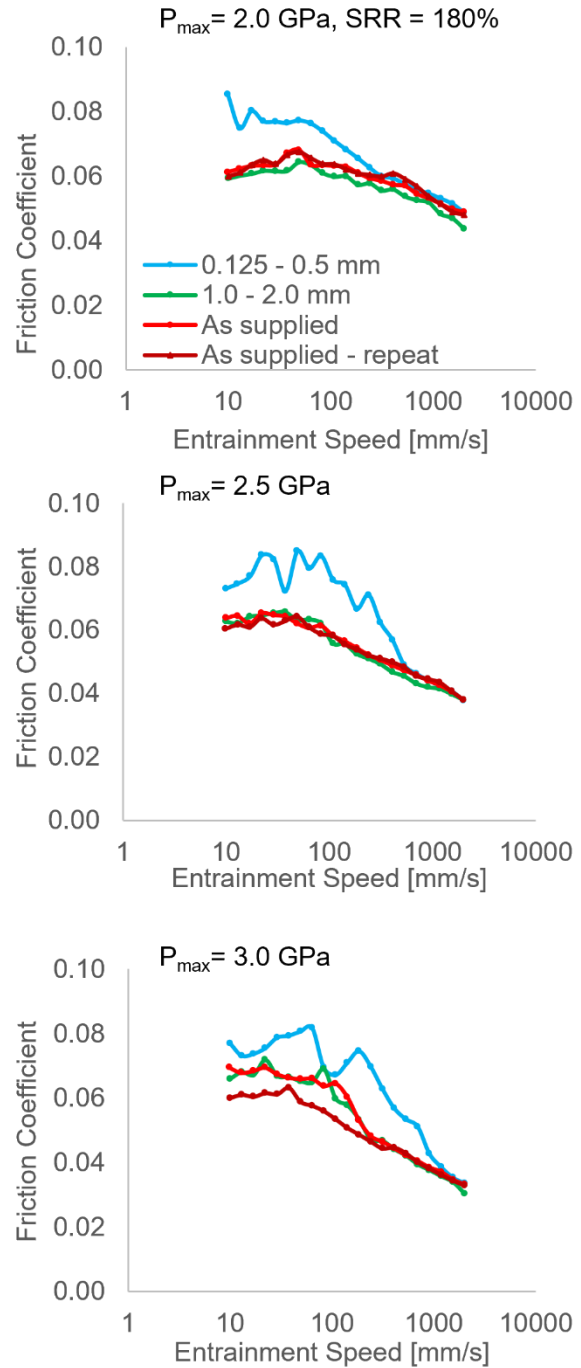


Figure 4-9: ETM friction with the sodium soap (78% FA content) of different grain size distribution ("as supplied", 0.125 – 0.5 mm, 1.0 – 2.0 mm), a) traction curves, b) 'Stribeck' curves.

The results for the low FA content calcium soap (35%) are presented in Figure 4-10. The key observation here is that especially at high sliding speeds and higher pressures of 2.5 and 3.0 GPa, there is no significant difference in friction achieved between the different grain size distributions.

Considering the traction curves (Figure 4-10a), a very similar friction behaviour is seen between all fractions at all pressures despite the low repeatability at low SRRs of this calcium soap, see chapter 4.1. In particular at high SRRs again, identical COFs are observed.

In case of the Stribeck curves (Figure 4-10b), the fraction sizes seem to have different effect on friction depending on the pressure. At 2.0 GPa, significant discrepancies are seen between the fractions. At lower speeds, friction seems to be highest for the smallest fraction with friction decreasing with increasing speed, whereas the other two fractions start with lower friction and increase similarly with speed. The “as supplied” fraction increases continuously, while the intermediate fraction (1.0 – 2.0 mm) seems to reach a level at higher speeds. The smallest fraction shows a sharp increase at high speeds and seems to follow the behaviour of the “as supplied” fraction, here. In all cases, the friction curves are not as smooth compared to the sodium soap.

At 2.5 GPa and 3.0 GPa, the differences between the “as supplied” and “1 – 2 mm” fraction are only minimal throughout the entire test. The smallest fraction “0.125 – 0.5 mm” deviates from the rest by continuously resulting in higher friction up to speeds of around 500 mm/s, showing same characteristics as the sodium soap above (Figure 4-9b).

In summary for the calcium soap (35%), at high SRR, high speeds and high pressures of 2.5 GPa and 3.0 GPa, the different fractions do not hugely affect the coefficient of friction. Only at lower pressures of 2.0 GPa, disparities in friction are observed. This could perhaps be due to an easier crushing or plastic deformation of the soap particles and therefore, faster film formation at high pressures, or due to a temperature effect.

a) Traction Curves with Ca-soap (35%) b) 'Stribeck' Curves with Ca-soap (35%)

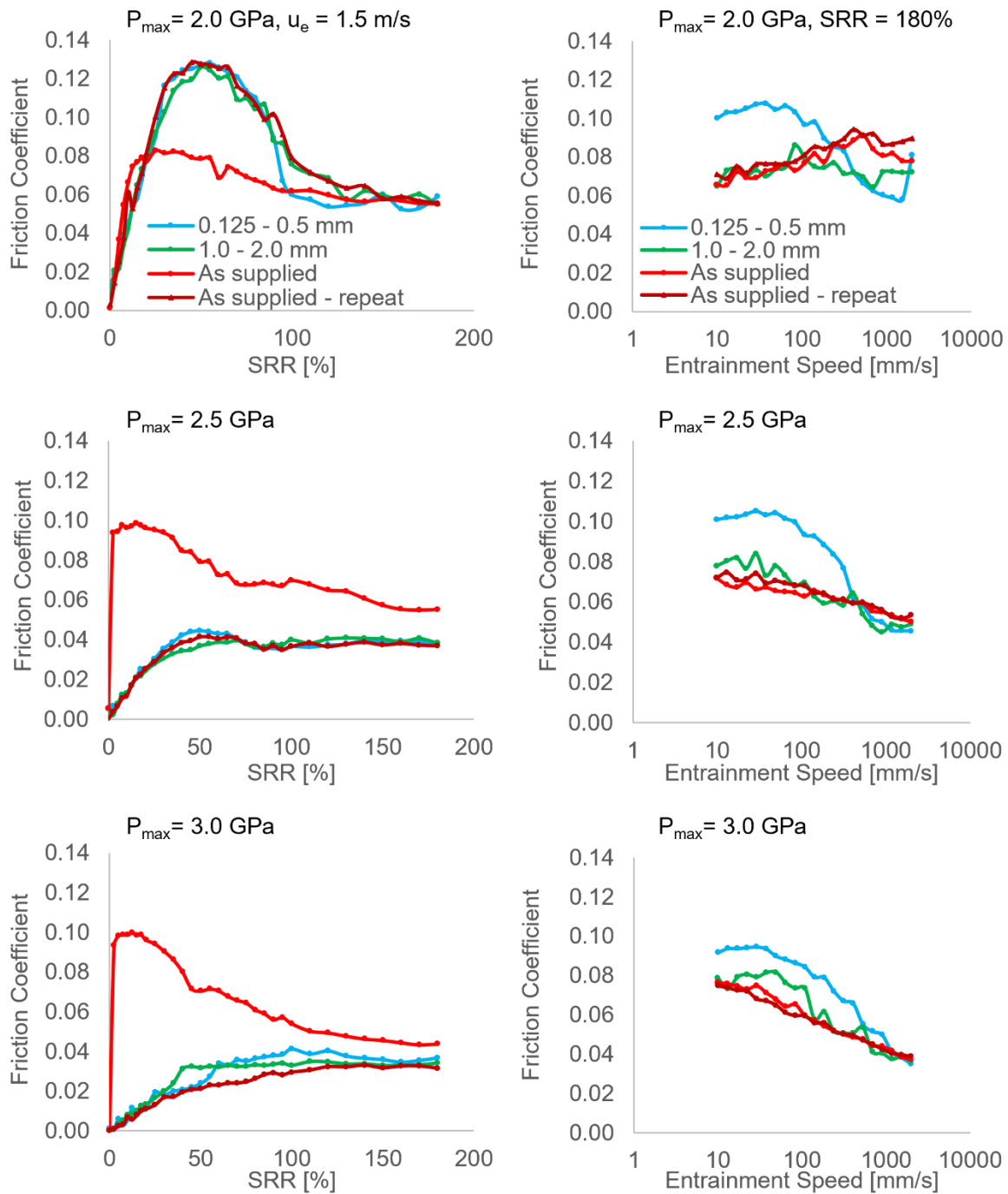


Figure 4-10: ETM friction with the calcium soap (35% FA content) of different grain size distribution ("as supplied", 0.125 – 0.5 mm, 1.0 – 2.0 mm), a) traction curves, b) 'Stribeck' curves.

4.8 The Effect of Surface Treatment of Steel Discs

All tests above were performed with case carburised C15E steel discs as specified in chapter 3.2.2. The main reason for this was to increase the surface hardness of the discs to avoid rapid plastic deformation and thus, to ensure controlled contact conditions throughout the test. However, in wire drawing, the wire material is of course not case carburised but is soft and undergoes huge plastic deformation. Thus, the effect of the surface treatment of the steel discs (case carburised vs. annealed) on friction has been studied for better comparison with the real application.

Figure 4-11 shows the comparison of measured ETM friction between the case carburised C15E discs and the discs in their initial annealed state. For each soap, there is little to no difference in COF between tests done with case carburised and annealed discs. This supports the use of the case carburised discs for the scope of this thesis.

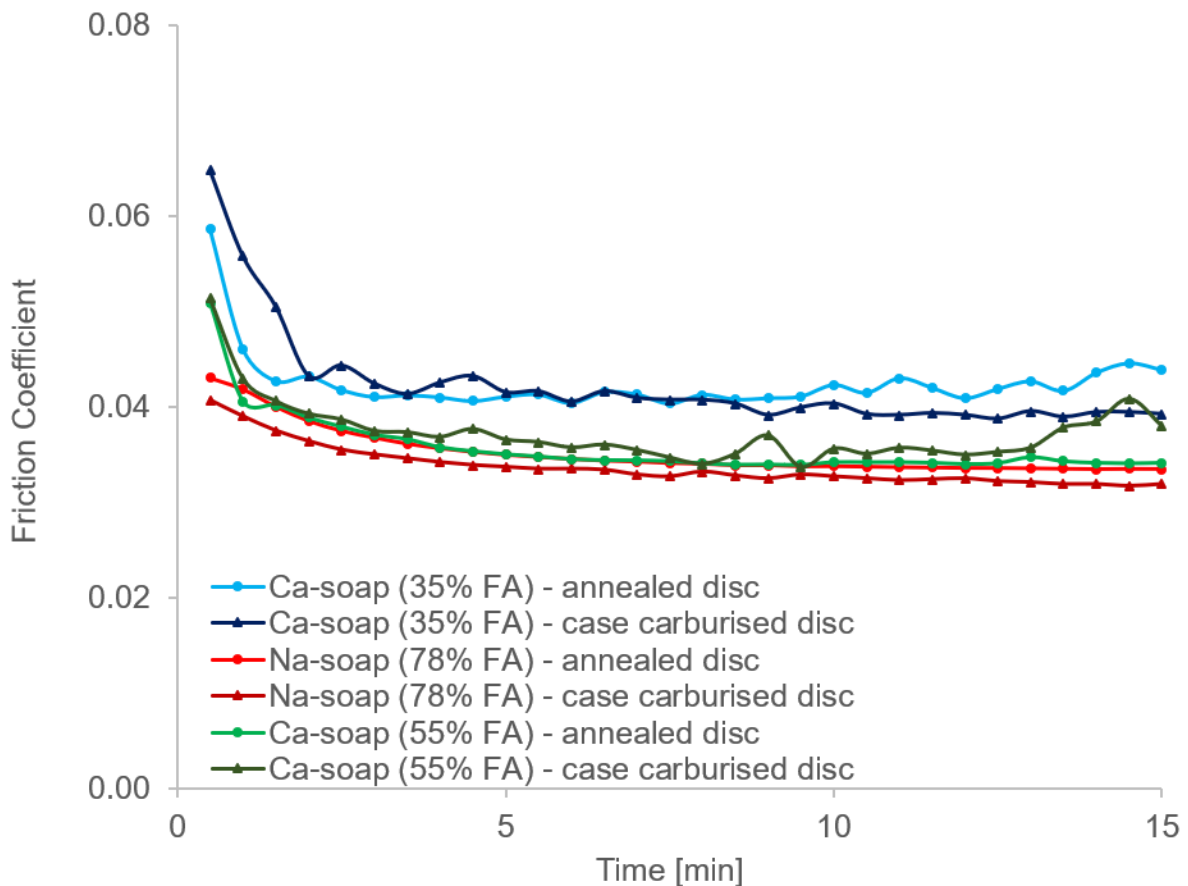


Figure 4-11: Friction with case-carburised vs. annealed steel ETM discs for each soap ($u_e = 1.8$ m/s, $SRR = 180\%$, $P_{max} = 2.5$ GPa).

4.9 Discussion of Frictional Behaviour

The results show that at high sliding speeds and high pressures, soap friction can be as low as 0.03 which is comparable to friction with lubricating oils under full film elastohydrodynamic conditions. The fact that friction decreases with increasing sliding speed and pressure is practically significant since the real wire drawing involves high pressures, to induce plastic deformation of the wire, and very high sliding speeds, to improve the productivity. The trend is also likely to be indicative of the prevailing mechanisms that may be responsible for the low soap friction. In particular, high sliding speed and high-pressure conditions lead to high contact flash temperatures, and it may be that this high in-contact temperature is the necessary condition for soaps to exhibit the low friction observed. This could be simply because the soap particles become softer and easily sheared at high temperature, but the very low values of friction suggest that perhaps soap has transitioned to a fluid-like or gel state at these temperatures and is consequently able to form a low shear lubricating film, at least intermittently. With this in mind, the effect of temperature on the soaps and friction is very interesting and will be analysed in chapter 8.

The decreasing friction with increasing log speed behaviour of soaps (Figure 4-2) is reminiscent of the mixed lubrication regime part of a typical Stribeck curve for lubricating oils. Notwithstanding the fact that this work is dealing with solid, powdered soaps here, this may suggest a mixed-lubrication type behaviour, similar to the findings of Levrau (54). In contrast, most literature assumes that full film hydrodynamic or quasi-hydrodynamic lubrication occurs in wire drawing with dry powdered soaps when a pressure-die is used (38,46,47).

The effect of shear stress decreasing with increasing speed (Figure 4-4) is similar to what was observed for calcium stearate monolayers by Briscoe *et. al.* (69). This could again be linked back to the stearic acids of the soaps which show a temperature-dependent decrease in shear strength (70). At lower speeds, a significant difference between pressures is observed, where shear stress increases linearly with increasing pressures (Figure 4-5) similarly to studies on calcium stearates (71). At high speeds, these discrepancies between pressures vanish and shear stress is the same. Only for the 2.0 GPa pressure of the calcium soap with low fatty acid content, shear stress increases further but seems to decrease only after around 500 mm/s. This 'delay' in

decrease could again suggest that an increase in temperature is crucial to reduce friction and a combination of high pressure and high speed would lead to lowest shear stresses.

The friction tests in this study show that the sodium-based soap (which also had a relatively high fatty acid content of 78%) consistently leads to lower friction coefficients at high speeds and pressures than the calcium-based soap tested (lower fatty acid content of 35%). This appears to be in line with wire drawing practice where sodium soaps are typically used wherever possible, especially at high speeds, and calcium soaps are generally employed in situations where sodium soap performance becomes inadequate³. This similarity in trends between current lab experiments and real wire drawing practice gives some confidence that the ball-on-disc tests are comparing the relevant factors in soap wire-die lubrication.

The tested soaps have a very different chemical composition other than the metal ion content alone, in particular, the fatty acid content is very different. In fact, in all tests conducted, the selected calcium soap with medium 55% FA content showed more resemblance in friction behaviour with the sodium soap with even higher FA content of 78%, in particular at high speeds and high pressures. Therefore, any differences in friction behaviour of soaps cannot be attributed to the metal itself but are also influenced by other compounds present in each soap formulations, not least the fatty acid content. Direct comparison of pure calcium and sodium stearates (Figure 4-6) confirmed that friction behaviour between these two stearates is quite similar. However, the friction trends obtained with the formulated 55% FA content calcium soap also indicated some fluctuations or instabilities with the soap, more comparable to the low FA calcium soap (35%), see for example Figure 4-3. Hence, the frictional performance depends strongly on the combination of soap metal and fatty acid content.

One additional parameter that could affect friction, was the grain size of the particles. The tests conducted with very small particles, however, indicated very little differences in COFs compared to the unfiltered “as supplied” fraction at high speeds and

³ Implicit industry know-how, personal communication by Stijn de Pauw, Bekaert

pressures. In fact, the only disparities were seen at lower speeds where the lowest particles showed highest friction. This is contrary to the findings of Byon *et. al.* (55) who describe a full-film formation with smaller particles by covering the entire wire surface better. This contradiction could be due to the difference in experimental set-up of the ETM. In the ETM, the soap particles are constantly compacted by the ETM ball which could lead to a lower grain size from the beginning. In addition, the particles entering the contact could be a mixture of new particles as well as particles from the previous revolution which could improve lubrication. Nevertheless, Byon *et. al.* (55) also showed very comparable trends between the different grain sizes during drawing stress measurements which aligns with the ETM friction measurements.

Other than indications as to the potential mechanisms of soap lubrication, which will be discussed further later in this thesis, the major contribution of this chapter is the actual soap friction values. Such data simply did not exist before but is crucial in improving the wire deformation models used to optimise the industrial wire drawing processes. In fact, the relevant models have hitherto used much higher COFs between 0.05 and 0.10 (55,59), so the simple friction data presented here is of immediate benefit in improving these models.

4.10 Summary of the Key Findings of the Soap Friction Tests

This study investigated the frictional behaviour of typical sodium- and calcium-based wire drawing soaps using the ETM ball-on-disc tribometer. The key findings of the friction tests are summarised as follows:

- The ETM tribometer with an addition of a scoop can be successfully used to directly measure friction with wire drawing soaps under representative conditions.
- The tested sodium and calcium soaps produced very low friction coefficients of 0.03 and 0.04 at high sliding speeds and high pressures.
- Soap friction generally decreased with increasing sliding speed and pressure. Soap friction was somewhat erratic at lower sliding speeds but was very steady and low at higher sliding speeds.
- Soap formulation is important for friction. Friction tests with pure sodium and

calcium stearates showed that the fatty acid content in particular seems to have a crucial influence on the soap frictional behaviour; the higher fatty acid content in formulated soaps resulted in lower COFs at high speeds and high pressures.

- A borax coating on the disc specimen surface led to lower friction at the start of the test. After a few minutes, friction increased to that of non-coated discs, perhaps suggesting that the borax coating is quickly removed.
- Different particle size distributions of soap powders did not show significant differences in friction.
- Case-carburising the discs to avoid large deformations during the ETM tests did not affect friction measurements.

The ETM experiments conducted give a good quantitative indication of frictional behaviour with dry wire drawing soaps. To better understand the observed friction trends, it is necessary to understand the nature of the lubricating films which is done in the next chapters.

5 Post-test Analysis of Deposited Soap Layers

The soap films formed on the ETM specimens during the tests described in chapter 4 were studied post-test to enhance the understanding of the nature of soap films. This chapter describes this post-test analysis of the soap deposited layer on the ETM specimens using confocal microscopy, Raman spectroscopy, as well as SEM-EDX microscopy. A brief analysis of the wear track is also described. Parts of this chapter have been published by the present author in the open access paper (41).

5.1 *Optical Analysis of Deposited Film on Rubbing Track*

The ball and disc specimens were analysed after the ETM tests described in Chapter 4. Figure 5-1 and Figure 5-2 show example confocal microscope images of typical disc rubbing tracks after Traction and Stribeck curve tests from chapter 4 respectively, with sodium and calcium films, and the corresponding topography of the same areas. Confocal microscope has the ability to record both the image and topography at the same time. Together, images and surface topography measurements indicate the presence of a residual film in the rubbing track on the disc surfaces with both sodium and (35% FA) calcium soap. With the (35% FA) calcium soap, some soap lumps are also seen on the disc track (see Figure 5-2b). For the (55% FA) calcium soap, however, no residual film was seen on the rubbing track (see Figure 5-1c and Figure 5-2c). This suggests that the (55% FA) soap either did not form any films during these particular tests or that it formed somewhat weaker films which adhered less on the surface and were removed due to the shearing during rubbing tests.

After the long-term tests (1 hour of rubbing at 2.0 GPa, $u_e = 1.8$ m/s, $SRR = 180\%$), a residual film is present for all three soaps (Figure 5-3). For all three soaps, the films seem to be inhomogeneous, in particular for the sodium soap, the rubbing track is only partly covered with a soap layer (Figure 5-3a). The (35% FA) calcium soap shows again some bigger lumps most likely originating from compacted soap powder (Figure 5-3b). For the (55% FA) calcium soap, a residual film can be seen which covers most of the rubbing track. Only in the central area of the wear track, the soap film was removed.

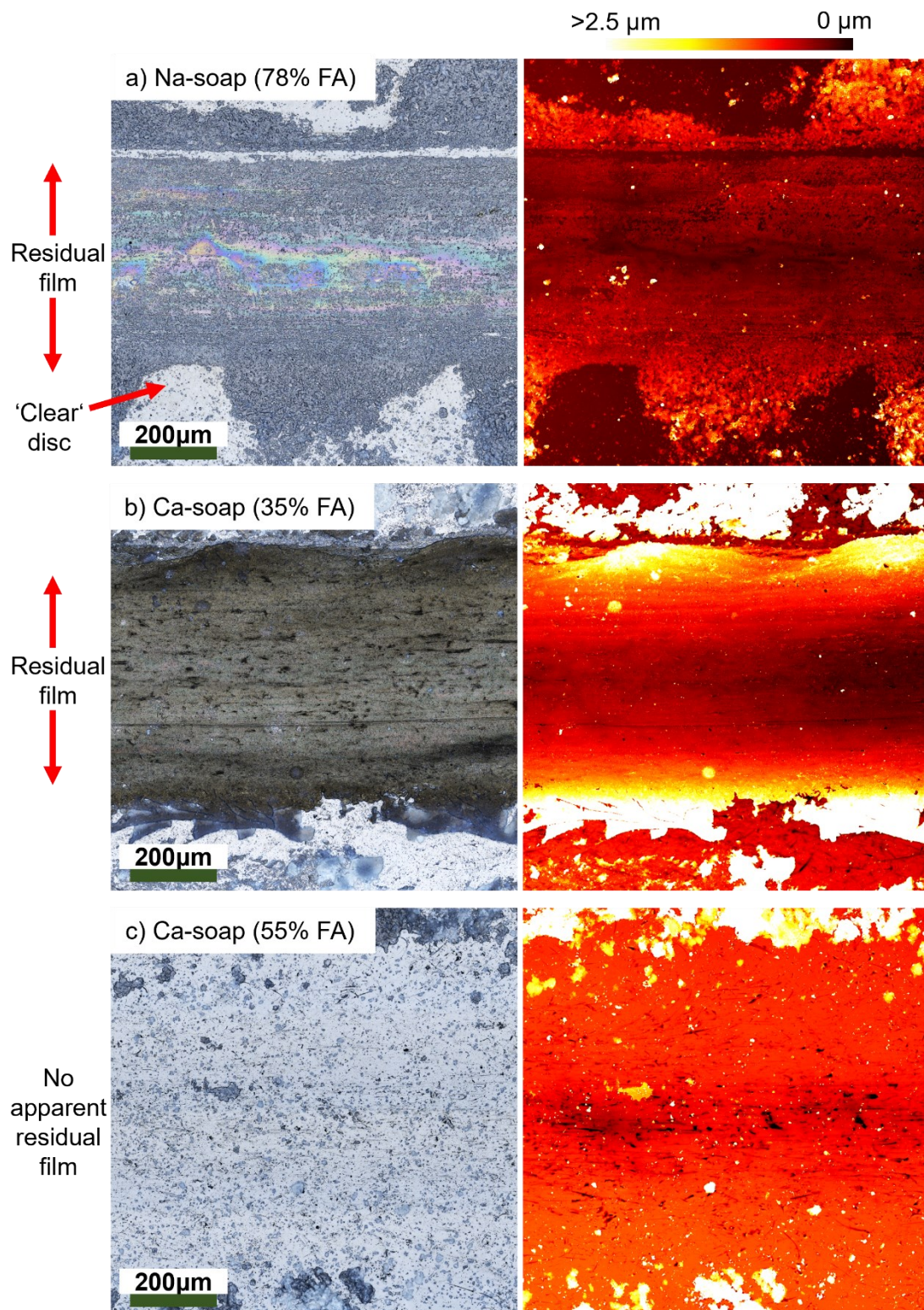


Figure 5-1: Example images (left) and corresponding surface profiles (right) of typical residual soap films in rubbing tracks on C15E disc surfaces post ETM tests with varying SRR (measured with confocal microscope).

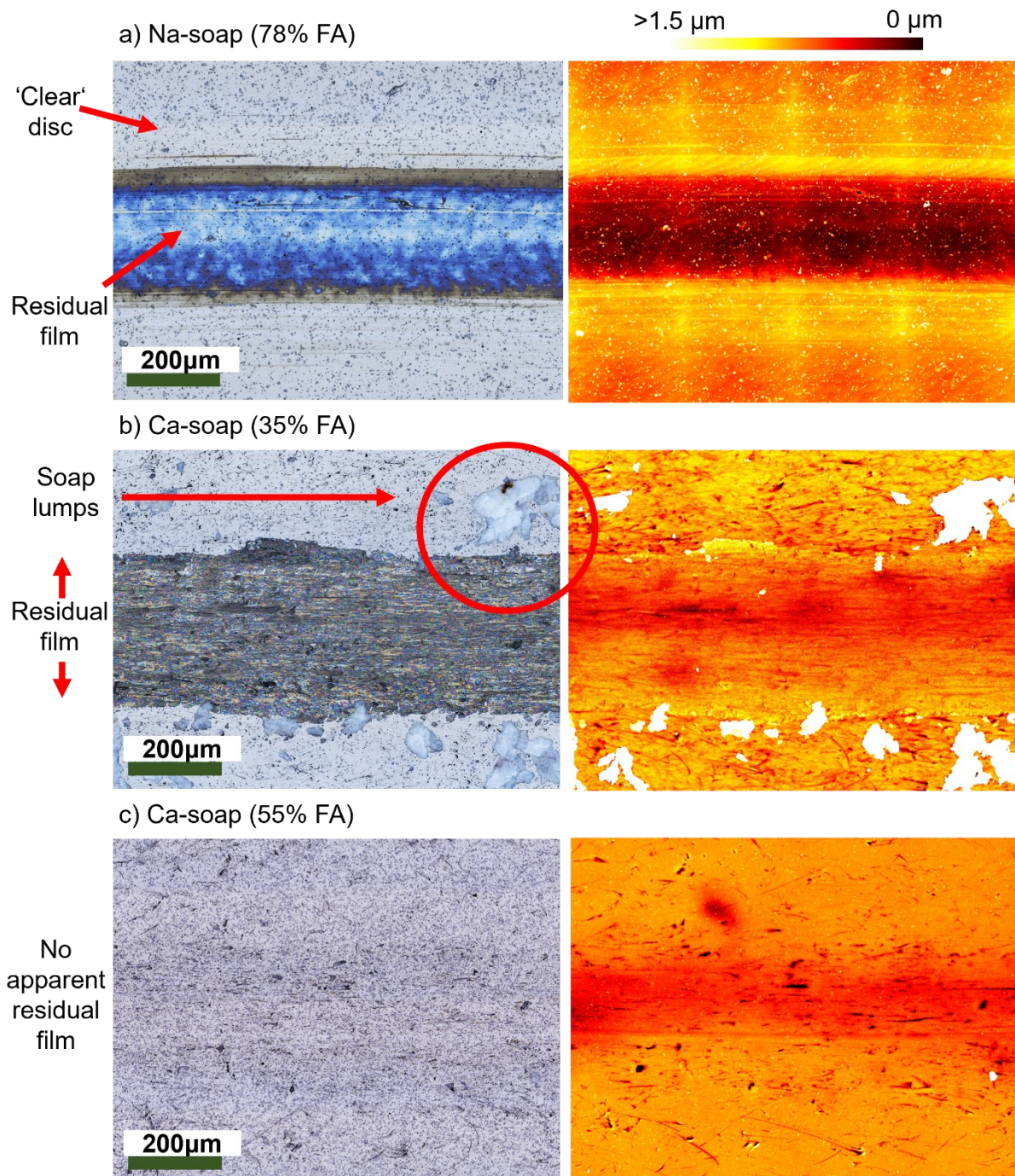


Figure 5-2: Example confocal images (left) and surface profiles (right) of typical residual soap films in rubbing track on C15E disc surface post ETM tests with varying speed (measured with confocal microscope).

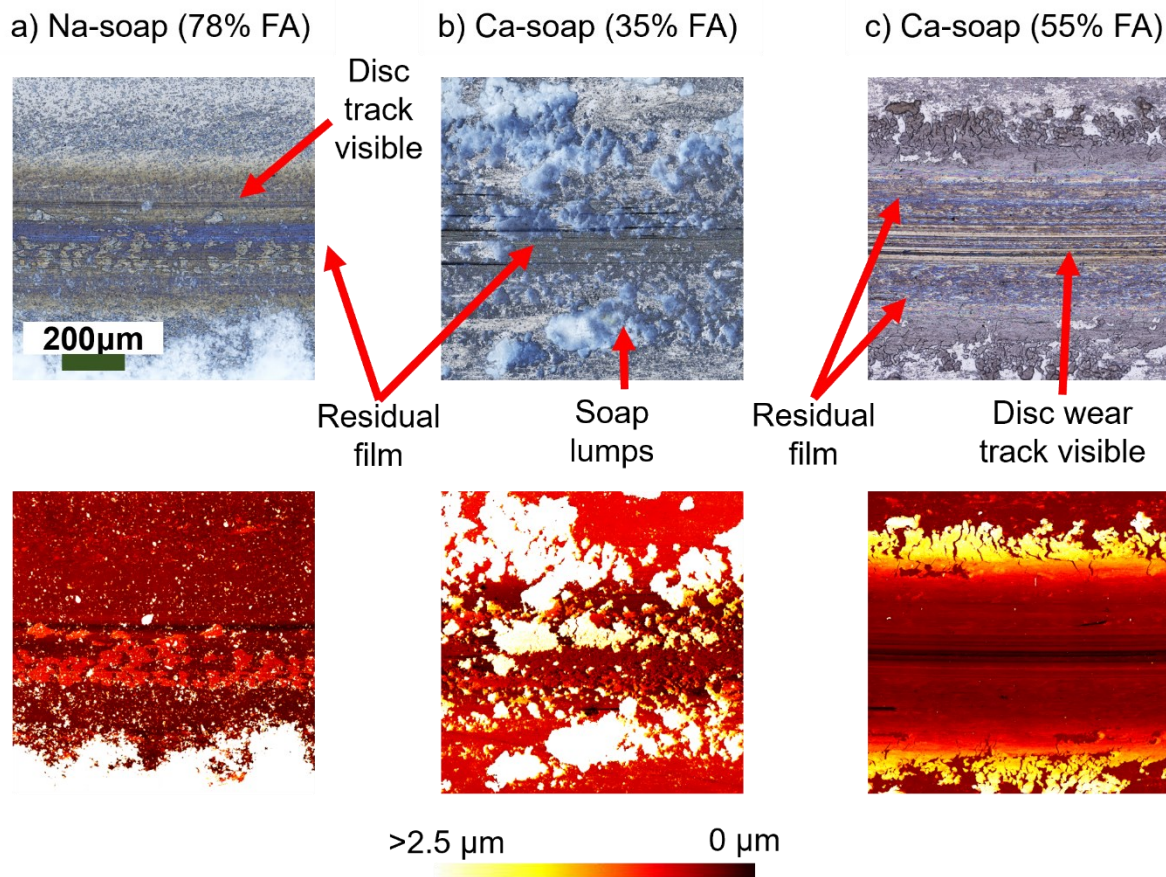


Figure 5-3: Example confocal images (top) and corresponding surface profiles of the same area (bottom) of typical residual soap films in rubbing tracks on C15E disc surface post long-term ETM tests, 1h at 2.0 GPa, $SRR = 180\%$, $u_e = 1.8$ m/s (measured with confocal microscope).

The film thickness of the soap layers could be estimated from the differences in height of the undisturbed film surface and height of a disc area where the residual film was deliberately removed (as described in the test procedures in chapter 3.2.3). The average film thickness recorded in this way in the rubbing track from 20 measurements on a single disc for each soap are shown in Table 10. Relatively thick residual films in the rubbing track were measured for both sodium and calcium (35% FA) soap, with those for sodium reaching 300-500 nm and those for the low FA (35% FA) calcium being much thicker at about 1.3 μm . After the long-term test (1h, 2.0 GPa), the residual calcium (35% FA) soap layer beneath the bigger soap particles is slightly thinner with a thickness of around 0.5 μm . For the medium FA (55%) calcium soap, no residual film thickness could be detected with the confocal microscope after the tests of varying SRR and varying speed, confirming the observations above where no films were

evident in confocal images in this case. However, after the long-term friction tests (1h at 2.0 GPa, 1.8 m/s, 180% SRR), the residual film with this soap (55% FA calcium) was estimated to be around 0.4 μm .

Table 10: Average measured thicknesses of residual soap films within the disc rubbing track after friction tests (reported values are measured as described in section 3.2.3)

Type of friction test	Average film thickness		
	<i>Na-soap (78% FA)</i>	<i>Ca-soap (35% FA)</i>	<i>Ca-soap (55% FA)</i>
<i>Varying SRR</i>	0.5 μm	1.3 μm	Not measurable
<i>Varying speed</i>	0.3 μm	0.4 μm	Not measurable
<i>1h – 2.0 GPa</i>	0.5 μm	0.5 μm	0.4 μm

Figure 5-4 shows confocal images of the rubbing track on WC ball that correspond to the disc images in Figure 5-1 and Figure 5-2. Once again, the presence of residual soap film is evident in the confocal images, with apparent differences in morphology of the film between calcium and sodium films. The calcium (35% FA) film was more prominent in the rubbing track whereas for the sodium soap, the film was removed from the rubbing track, in particular during the tests with varying speeds. With the calcium soap (55% FA), a deposited film seems to be present for the tests with varying SRRs, whereas for the varying speeds test, no residual film is seen on the ball after the tests.

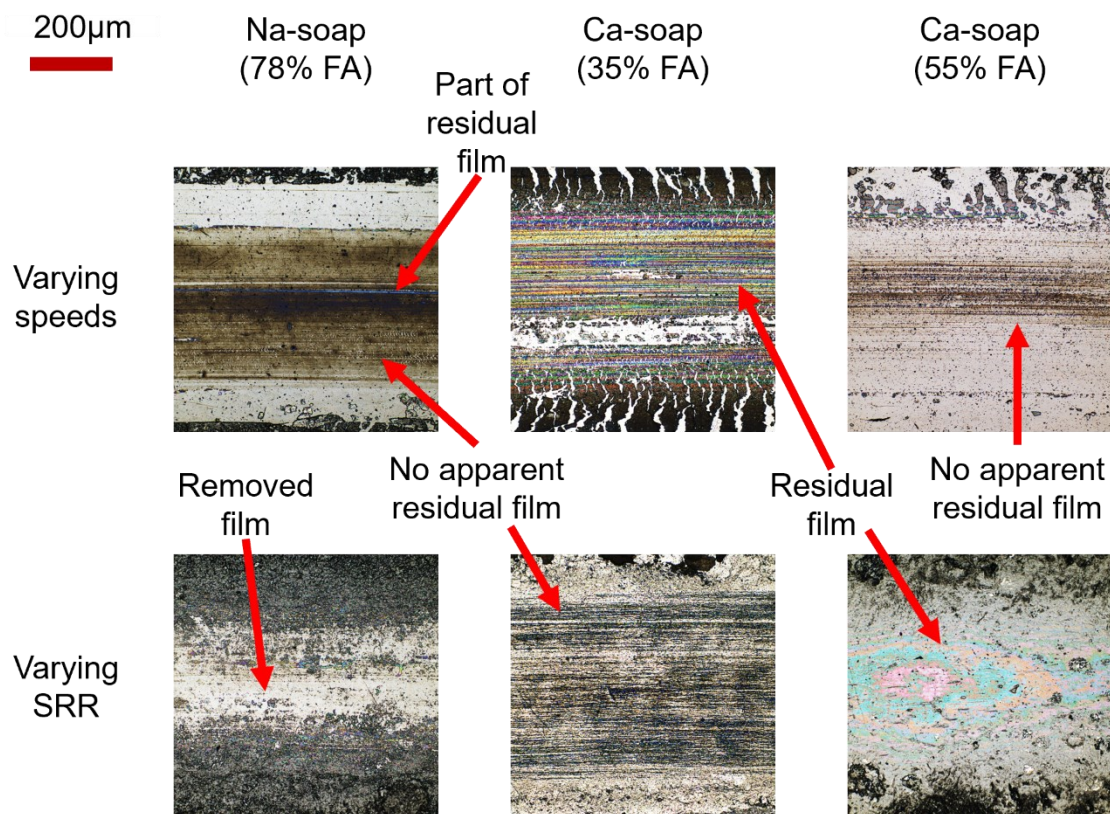


Figure 5-4: Example confocal images of rubbing track on WC ball after ETM tests.

5.2 Chemical Analysis of Soap Deposited Films in Rubbing Track

To get further information of the chemical composition of the deposited films, SEM-EDX measurements were conducted. However, since the three types of tested soaps have very different fatty acid content (see Table 7) and therefore, different amount of the respective metal content, the quantitative information on the amounts of different compounds present in the deposited films from the three soaps cannot be compared directly. The films formed with the (35% FA) calcium soaps have a high calcium content in the rubbing track with about 3.4 – 6 wt.% Ca on average over all tests conducted. The (78% FA) sodium film has a low sodium content of 0.9 – 2.2 wt.% on average. With the higher fatty acid content of 55% calcium soap, the produced film has also a low calcium content of about 1.1 – 2.5 wt.%.

Raman spectroscopy was used to study the composition of the deposited films. In each case, the Raman spectra of the deposited film were compared to Raman spectra of the corresponding soap itself as well as that of a clean disc surface. Raman spectra

were taken on four different areas on each disc and below figures (Figure 5-5, Figure 5-6, Figure 5-7) show spectra of typical observations for each test and soap.

Sodium film Raman spectra are shown in Figure 5-5. Spectra for fresh C15E ETM disc and soap powder are shown for comparison. As the spot size of the laser is small (around $0.7\ \mu\text{m}$), measurements were taken on the homogeneous regions of the deposited film to ensure that the obtained spectra are as representative as possible of the entire film in the rubbing track. The sodium soap film formed on the disc specimens (red and blue line in Figure 5-5) show significant peaks at 2850 and $2880\ \text{cm}^{-1}$ which can be assigned to the C-CH₃ and C-H peaks of the soap (green line). The peak at $1300\ \text{cm}^{-1}$, which corresponds to C-C aliphatic chain vibrations, as well as the peaks at around $2430\ \text{cm}^{-1}$ are attributed to the sodium soap. The peak at $1450\ \text{cm}^{-1}$ can be assigned to CH₂ and CH₃ peak of the sodium soap. A weaker peak at around $1060\ \text{cm}^{-1}$ could be assigned to C-O-Na vibrations which again relates to the sodium soap. At 80 to $90\ \text{cm}^{-1}$ lattice vibrations can be observed on the disc specimens, being representative of the clean C15E disc (black line). However, an issue here is that the Raman scanning depth can be up to $10\ \mu\text{m}$, and therefore, it could have measured the signal from the steel disc. A broader peak between 600 and $900\ \text{cm}^{-1}$ is also seen on the disc with the sodium soap residuals which cannot be assigned to either the clean C15E disc or the pure sodium soap. These peaks may suggest the presence of iron oxides, e.g. Fe₃O₄ (72) and γ -Fe₂O₃ (73), and might suggest some changes to the chemistry of the produced film on the disc surface.

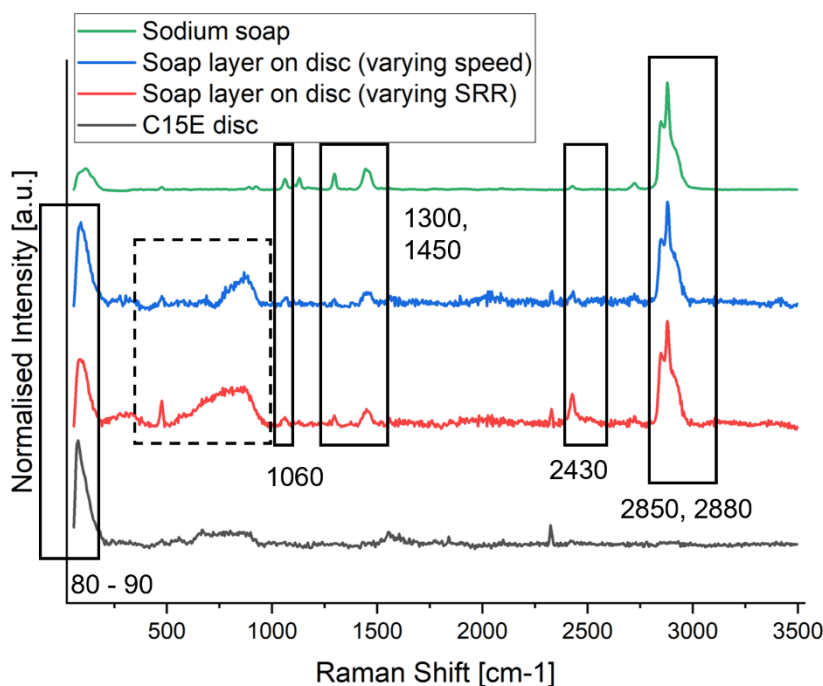


Figure 5-5: Typical Raman spectra of residual films on ETM disc specimens after tests with sodium soap. Spectra of original C15E disc and pure fresh soap are also shown for comparison.

Figure 5-6 shows the Raman spectra obtained from the (35% FA) calcium films. The deposited film layers on the disc specimens have very similar peaks to the (35% FA) calcium soap (green line in Figure 5-6). The peak around 140 cm^{-1} is attributed to lattice vibrations. The peaks around 400 cm^{-1} , 515 cm^{-1} and 640 cm^{-1} can also be assigned to the calcium powder, the latter originating from C-C aliphatic chain vibrations. A peak at 1080 cm^{-1} is seen which could be representative of the C-O-Ca vibrations of the calcium soap. The peaks at 1300 cm^{-1} and 1450 cm^{-1} correspond to C-C and CH_2 or CH_3 vibrations of the soap. The peak at around 2430 cm^{-1} could perhaps be an overtone (74) and is related to the calcium soap because no peak is seen on the clean disc at this Raman shift. In addition, C- CH_3 and C-H vibrations of the soap can be seen at a Raman shift of 2850 cm^{-1} and 2880 cm^{-1} , respectively. The similarity of peaks with the pure calcium soap powder suggests that the calcium soap in the residual film on the discs has not undergone significant changes from the original calcium soap powder.

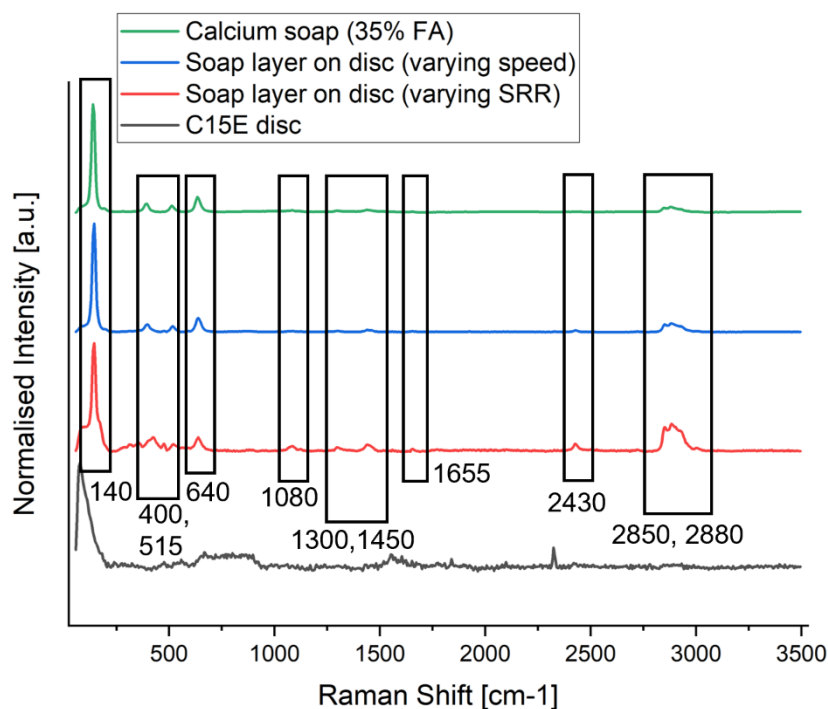


Figure 5-6: Raman spectra of residual films on ETM disc specimens after tests with (35% FA) calcium soap.

The Raman spectra for the medium fatty acid content (55% FA) calcium soap is shown in Figure 5-7. It is obvious that the peaks of the surface of the disc rubbing track are quite similar to that of the (55% FA) calcium soap. However, Figure 5-1c and Figure 5-2c shown above indicate no or very thin films with this calcium soap. Hence, the peaks recorded with the Raman spectra either imply a very thin film present on the rubbing track, or the sensitivity of the laser could have picked up the signal from residual soap particles that are also seen on the surface. In contrast, no peak seems to resemble the spectra from the clean C15E disc which again could indicate a very thin formed residual soap layer on the rubbing track.

The peaks at 340 cm^{-1} and 480 cm^{-1} can be assigned to the calcium soap, whereby the former originated from C-C aliphatic chain vibrations. The peak at 1300 cm^{-1} are also attributed to C-C chain vibrations from this calcium soap. The peak at 1455 cm^{-1} corresponds to CH_2 and CH_3 , and 1520 cm^{-1} and 1560 cm^{-1} are attributed to C=C bonds of the soap. The peaks at 1765 cm^{-1} and 1925 cm^{-1} also originate from the soap whereby the former corresponds to C=O vibrations. Like the other two soaps, a peak at 2430 cm^{-1} arises from the soap, as well as a peak at 2940 cm^{-1} corresponding to C-H bonds. The similarities in peaks imply that no significant chemical changes have occurred during the tests.

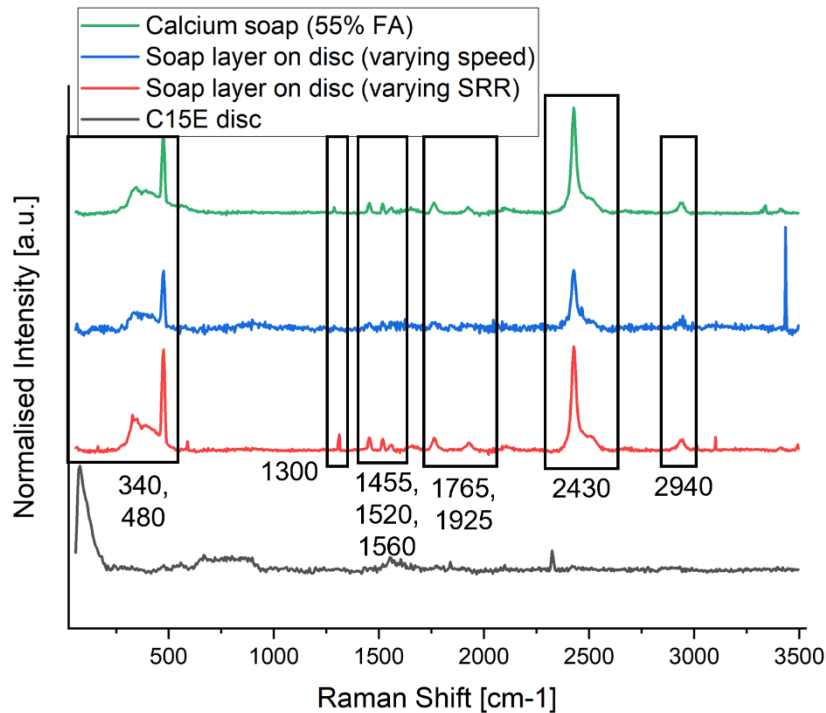


Figure 5-7: Raman spectra of residual films on ETM disc specimens after tests with (55% FA) calcium soap.

5.3 Durability of Formed Residual Soap Films

Chapter 4 showed that low COFs can be achieved with the tested soaps at high sliding speeds and high pressures. Relatively thick residual films were also formed on the discs, at least for the sodium-based soap and the (35% FA) calcium soap. Moreover, chapter 4.3 also showed that low friction behaviour can be achieved with soaps over a long period of time. So, a question that arises from this is whether or not these formed residual films persist under longer-term rubbing.

To investigate this, ETM tests were conducted over a period of one hour at fixed contact conditions, SRR of 180%, pressure of 2.5 GPa and entrainment speed of 1.8 m/s (Step 1). In order to assess the durability of the residual soap films, at the end of this 1-hour test, the samples were removed from the machine, the soap powder was removed from the pot using a vacuum, the specimens with the residual film were fitted back in the pot (now with no fresh soap present), and the test restarted (Step 2).

The results of these tests are shown in Figure 5-8. Step 1 is the initial step of 1-hour

where soap films were produced, and Step 2 is the subsequent test where the soap was removed from the ETM pot. The results of this Step 1 were already shown and described above in Figure 4-3 (long-term tests in chapter 4.3). Low friction is achieved with both sodium and low fatty acid content (35% FA) calcium soap. The medium fatty acid content (55% FA) calcium soap shows some instabilities but nonetheless, rather low COFs.

In Step 2, where the soap was removed and the test restarted, in the case of sodium soap a slightly higher COF is measured than before stopping, namely between 0.04 and 0.07. Friction is also a lot more variable than when the soap was in the pot in Step 1. After about 180 seconds of rubbing in Step 2, the COF sharply increases, jumping from 0.04 to about 0.1 in a matter of seconds. This likely indicates a rapid failure of the lubricating sodium film at this time resulting in an increase in metal-to-metal contact and the corresponding rise in friction.

The behaviour with both calcium-based soaps after the restart following soap removal is quite different, in that friction increases almost immediately (within 30s or so) after restart to about 0.10 and 0.13, for the (35% FA) and (55% FA) calcium soap respectively.

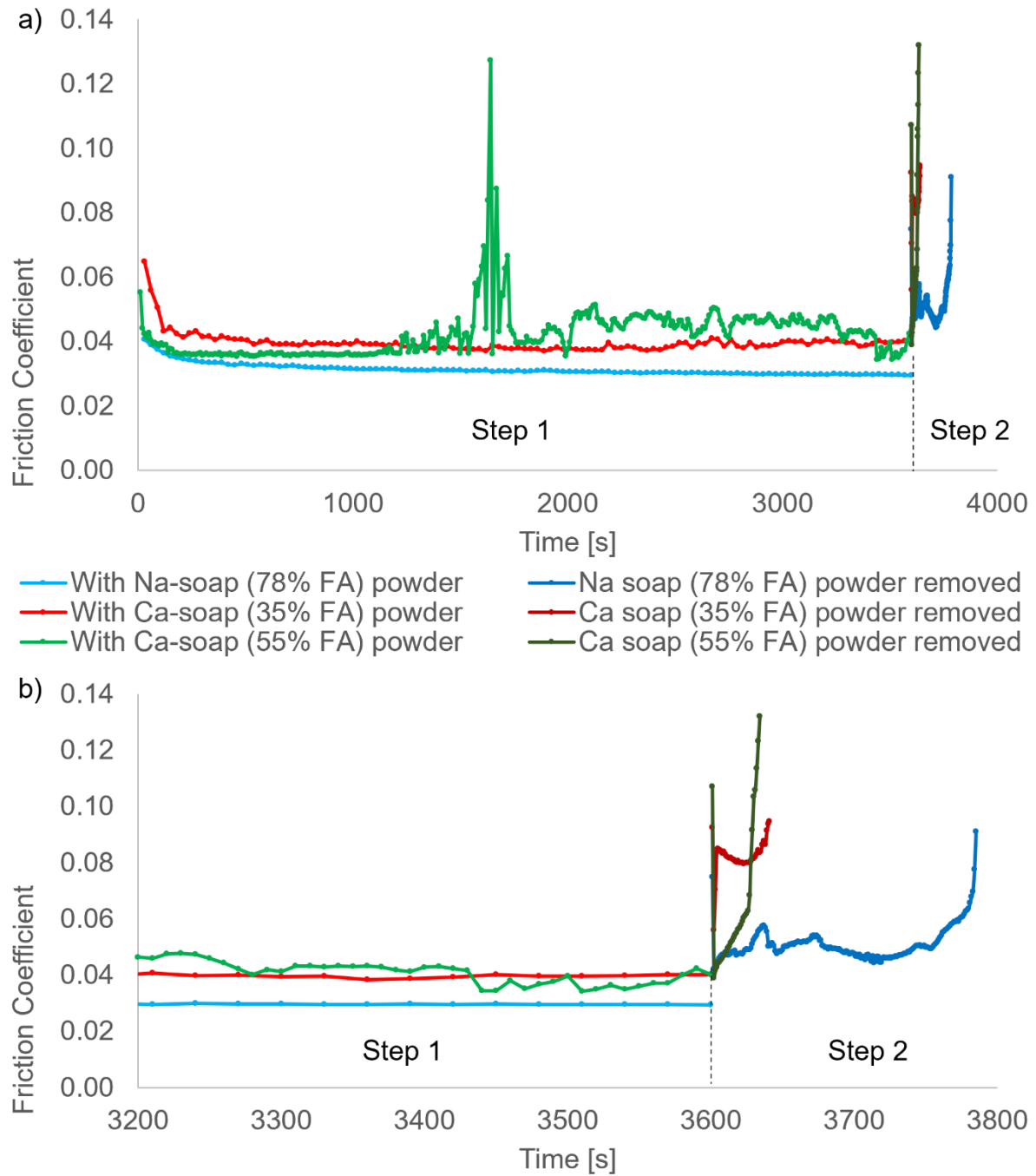


Figure 5-8: a) ETM friction with sodium and calcium soaps during a 1-hour test at $SRR = 180\%$, $P_{max} = 2.5$ GPa, $u_e = 1.8$ m/s, $T = 40^\circ\text{C}$ (Step 1). At 1 hour, the soap was removed from the pot and test restarted at same conditions (Step 2), b) zoomed-in plot showing end of Step 1 and whole of Step 2 ($3200\text{s} \leq t \leq 3800\text{s}$).

Confocal images of the soap films on the rubbing track at the end of each of the two steps (see Figure 5-8) are shown in Figure 5-9. At the end of Step 1, images show evidence of a residual compacted soap film in the rubbing tracks on the disc surfaces for all soaps; the morphology of films is quite different for the three soaps. Outside of the rubbing track, some soap particles are also accumulated in all cases, but these are less compacted than films within the track, and any soap layers here appear very porous. The presence of a residual film within the rubbing tracks coincides with the relatively low and stable friction coefficient for the sodium-based and low fatty acid content (35% FA) calcium soap as shown in Figure 5-8. In contrast, the images after Step 2 show that this film has now been removed from the rubbing track.

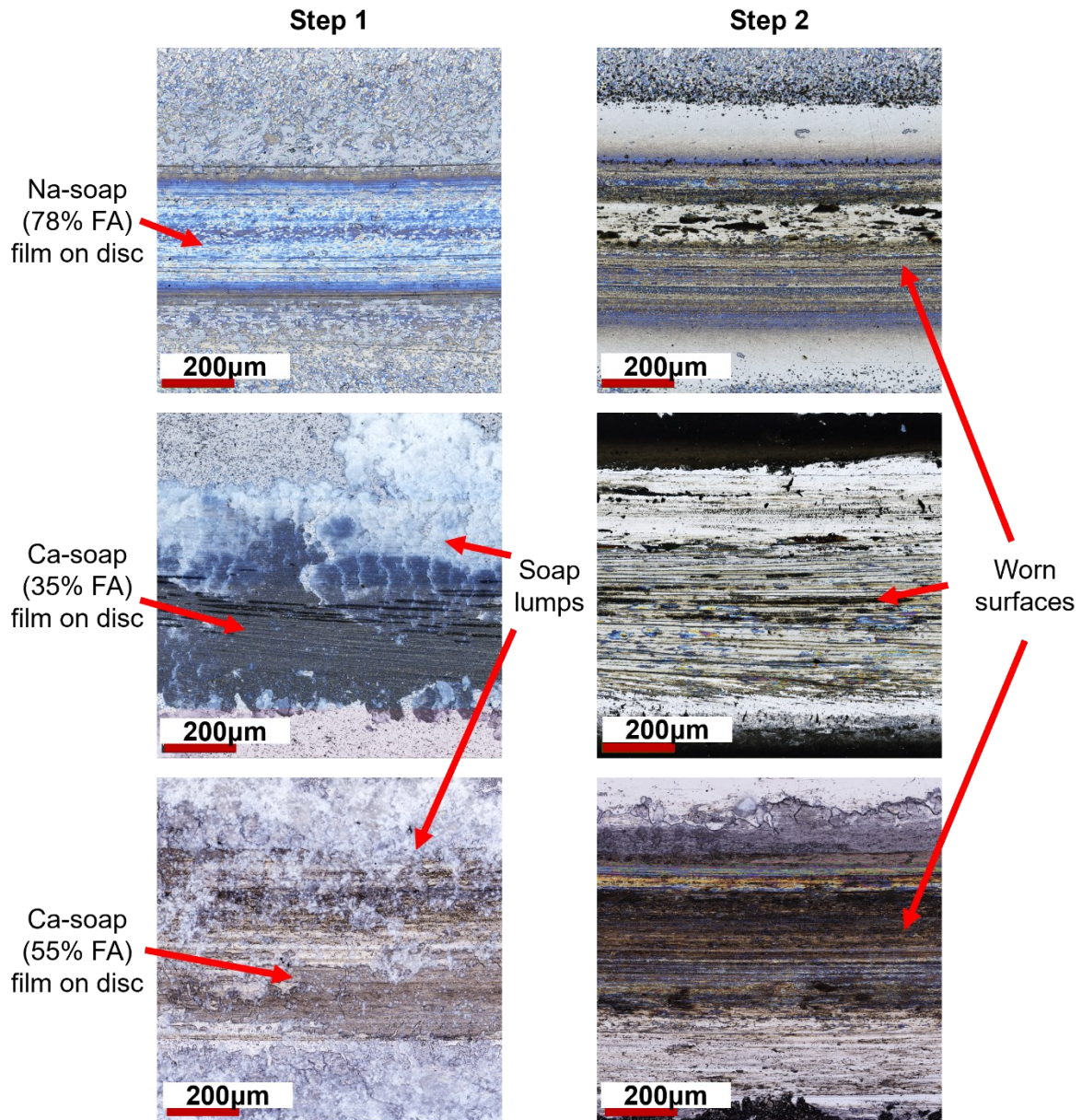


Figure 5-9: Confocal images of rubbing track on disc surfaces: after Step 1 in Figure 5-8 (1 hour steady state test) and after step 2 in Figure 5-8 (friction increase after soap removal); $SRR = 180\%$, $P_{max} = 2.5\text{ GPa}$, $u_e = 1.8\text{ m/s}$, $T = 40^\circ\text{C}$.

For the medium fatty acid content (55% FA) calcium soap, a thin, more inhomogeneous residual film is seen at the end of Step 1 which accounts for the instabilities seen in friction with this soap. After Step 2, the residual film was completely removed.

Furthermore, for all soaps the wear track after step 2 indicates significant damage, with torn, furrowed surface appearance that is very similar to typical scuffing damage that

has previously been generated in the ETM/MTM tribometers (75) or indeed real bearing surfaces, see (76). The increase in friction seen at this point (Figure 5-8) is also indicative of the onset of scuffing in the MTM (75). Rapid occurrence of scuffing, following the apparent failure of soap deposited films, is not at all surprising in this case given the high pressure and high SRR, with these conditions known to increase the risk of scuffing. These observations indicate that the deposited, probably compacted, soap layers in the rubbing track play an important role in soap lubrication. Soaps are able to produce low friction and protect the surfaces from wear well despite the very harsh contact conditions, as long as there is a continuous soap supply to the contact. However, any surface soap films formed are weak and rapidly fail when the soap supply is interrupted leading to a rapid rise in friction and scuffing damage.

5.4 Wear Analysis of ETM specimens

To assess the wear behaviour of the studied soaps, the surface of the post-test ETM disc specimens were first cleaned with isopropanol and then the rubbing track was inspected on the confocal microscope. Figure 5-10 shows some example images of the measured 2D profiles of the disc rubbing track after a test with varying entrainment speed for studied soaps. The corresponding wear measurement data is shown in Table 11. It is evident that the sodium soap produced more wear than the low fatty acid (35%) calcium soap, with the maximum wear depth being about 0.6 μm for sodium and 0.2 – 0.3 μm for the low FA (35%) calcium soap. In addition, the wear track from the sodium soap is relatively smooth but has ridges at either side of the track. In contrast, the low FA (35%) calcium soap generates rougher wear tracks. A profilometer was used to measure the surface roughness before and after the experiments. For the Stribeck tests (varying entrainment speeds at constant SRR of 180%) an increase of Rq value of 30 nm was measured for the sodium-based soap and 85 nm for this low FA (35%) calcium-based soap.

For the medium fatty acid content (55%) calcium-based soap, where almost no residual film was observed, the wear track is the deepest, with a value of about 0.8 μm . Some furrows that are well over 1 μm deep can be seen. An increase of Rq value of 90 nm is recorded here.

Similar wear trends were observed on discs after other tests with these soaps; images and measurements shown here are typical. Negligible amounts of wear were observed on all ball specimens, which may be expected given the very high hardness of WC, although some minor scratches were observed after tests with the calcium soaps.

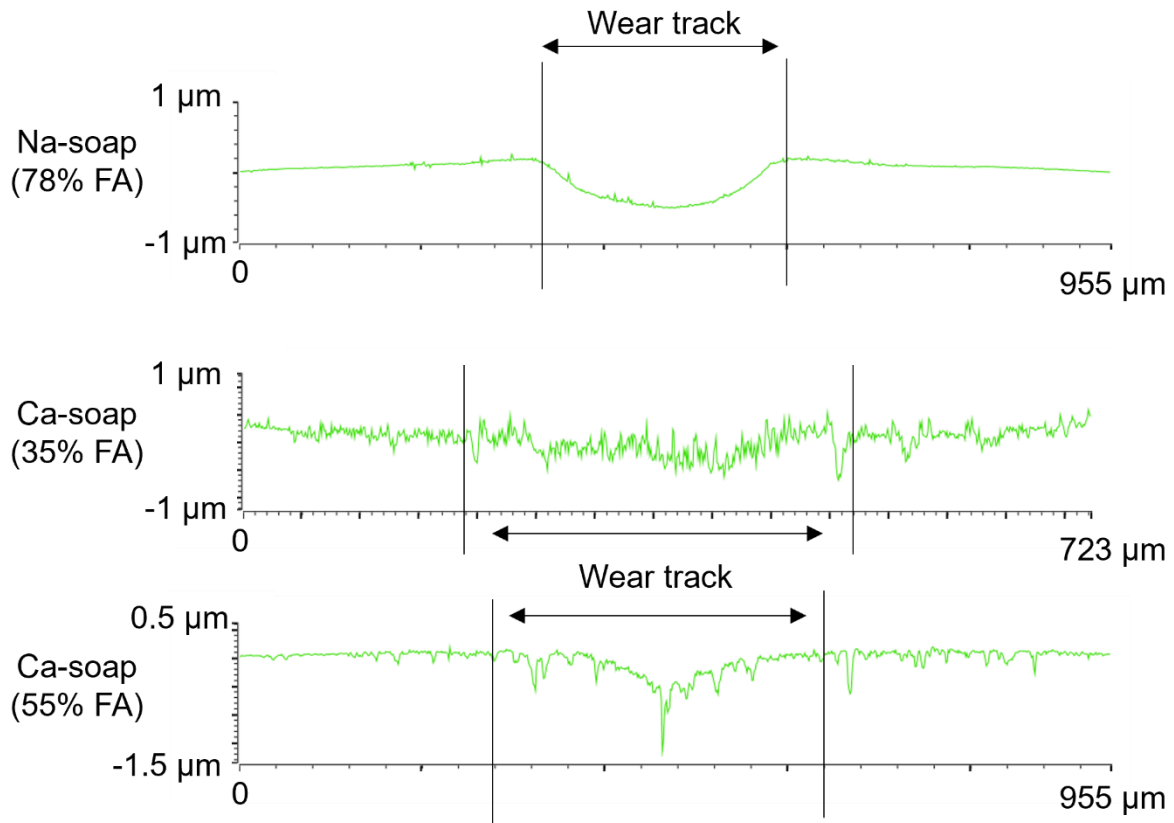


Figure 5-10: Typical profiles across rubbing track on ETM disc surfaces after tests with varying speeds with each of the three soaps as indicated after removal of any residual soap films using isopropanol. Profiles measured with confocal microscope.

Table 11: Wear on ETM disc surfaces after tests with varying speeds (corresponding data for Figure 5-10); note: wear depth measured with confocal microscope, R_q measured with stylus profilometer.

	Na-soap (78% FA)	Ca-soap (35% FA)	Ca-soap (55% FA)
Wear depth	0.6 μm	0.2 – 0.3 μm	0.8 μm
Increase in R_q	30 nm	85 nm	90 nm
Initial R_q	~ 15 nm	~ 15 nm	~ 15 nm

5.5 Discussion of Post-Test Analysis

An overview of the findings from this section is illustrated in Figure 5-11.

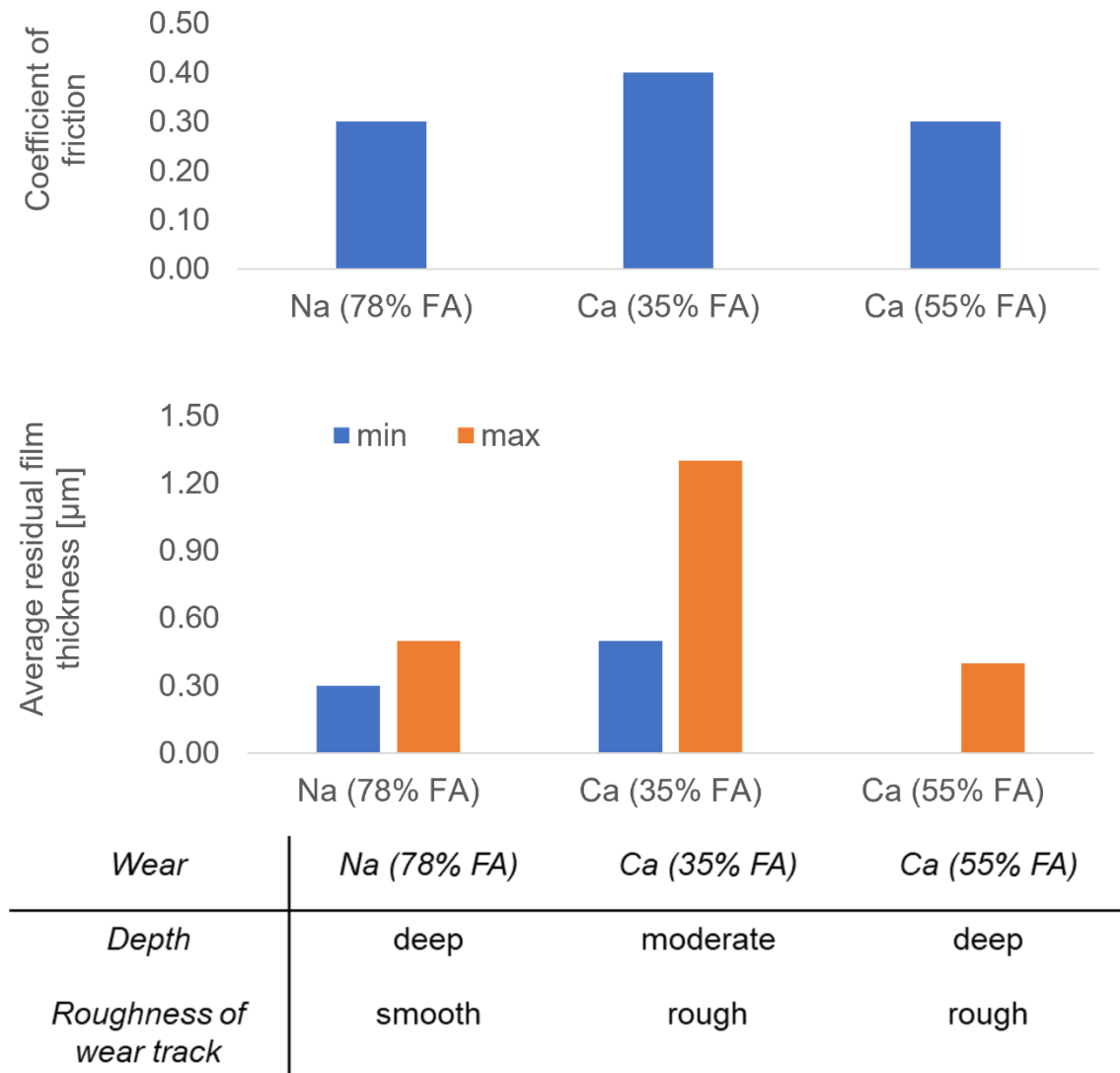


Figure 5-11: Summary of findings from this chapter (note: COF is shown for highest speed and highest pressure, $P_{max} = 3.0$ GPa, $u_e = 2.0$ m/s, $SRR = 180\%$; wear track assessment from tests above Figure 5-10).

The analysis of any deposited films on the rubbing track after the ETM tests are helpful to explain the low friction values achieved with soaps. Despite the very harsh test conditions at high sliding speeds and high pressures, the sodium-based and low fatty acid (35% FA) calcium soaps form relatively thick deposited films on the rubbing track after the ETM tests. The films have the same chemical composition as the fresh soap, so they do not undergo any major chemical changes during rubbing. These films are

rapidly removed when rubbed under same contact conditions but in absence of soap supply; their removal leads to a rapid increase in friction and occurrence of scuffing damage. Therefore, the presence of these deposited, compacted soap films appear to have a crucial role in effective lubrication provided by soaps.

In the case of the medium fatty acid content (55% FA) calcium soap, a residual film was clearly visible on the disc track only after the long-term steady conditions tests and not the 'Stribeck' and 'traction' tests. Nonetheless, in the latter cases, a transparent or very thin film could be present as the Raman spectra show very similar peaks as the soap powder.

The results and analysis presented in this chapter and chapter 4 show that the soaps are capable of providing low friction and wear, and that a continuous supply of soap into the contact is crucial in ensuring this. However, the mechanisms by which the soaps form any lubricating films during the rubbing process and the nature of these films (e.g. fluid vs. solid) has not yet been explored. Understanding this is crucial to be able to improve and model the soap lubrication performance and will be explored in the next chapter.

6 Direct Observations of Build-up of Soap Films During Rubbing

To better understand the lubrication mechanisms of powdered soaps at representative wire drawing speeds, the soap film in the contact was observed directly using a newly developed high speed optical ball-on-disc test rig. This chapter contains observations on the film formation and film behaviour up to high speeds (20 m/s), as well as on single soap particles passing through the contact. Most results shown in this chapter are with sodium soaps since it showed a more stable film behaviour, but some results for calcium soaps are included for comparison.

This chapter does not measure the actual film thickness, but it only describes them qualitatively. This is because of several issues with measuring soap films including the lack of knowledge of refractive index, the uncertain nature of films (transparent or non-transparent films) and their dynamic behaviour (as shown below).

6.1 *Direct Observation of Film Formation and Behaviour*

A set of experiments was conducted on the high speed optical EHD rig (EHD-HS by PCS) to observe how soap powders lubricate the contact. The exact procedure for these tests is detailed in Chapter 3.3. The test chamber, which contains the specimens in the rig is filled with soap powder so that the ball is fully immersed in the soaps. The observations are recorded as a high frame rate video, but for the purposes of this thesis they will be presented as a series of images representing the frames of interest taken from the original video.

The film formation of sodium soap over a short period of time at constant conditions of 0.5 GPa and 1.0 m/s entrainment speed is shown in a series of images in Figure 6-1. The SRR was set to -180% SRR so that the ball rotates faster than the disc and is able to draw soap powder from the reservoir into the contact. All tests conducted with the EHD-HS were performed at negative SRR (ball faster) for consistency. The inlet and outlet locations of the contact as shown in Figure 6-1 are the same in all subsequent images in this chapter.

From the colour changes in the images and their coverage of the contact area, it is clear that a soap film is building up and is forming a lubricating layer in the contact (Figure 6-1). In contrast to films formed by conventional standard oils, the soap films seen here are very dynamic and change in time even at steady state conditions; several features are apparent during the short time shown in Figure 6-1, with the film coverage and thickness changing to film collapse and apparent occurrence of starvation. Under these conditions, the soap film does not assume the conventional EHD horse-shoe shape, but at times, a constriction near the outlet can be observed.

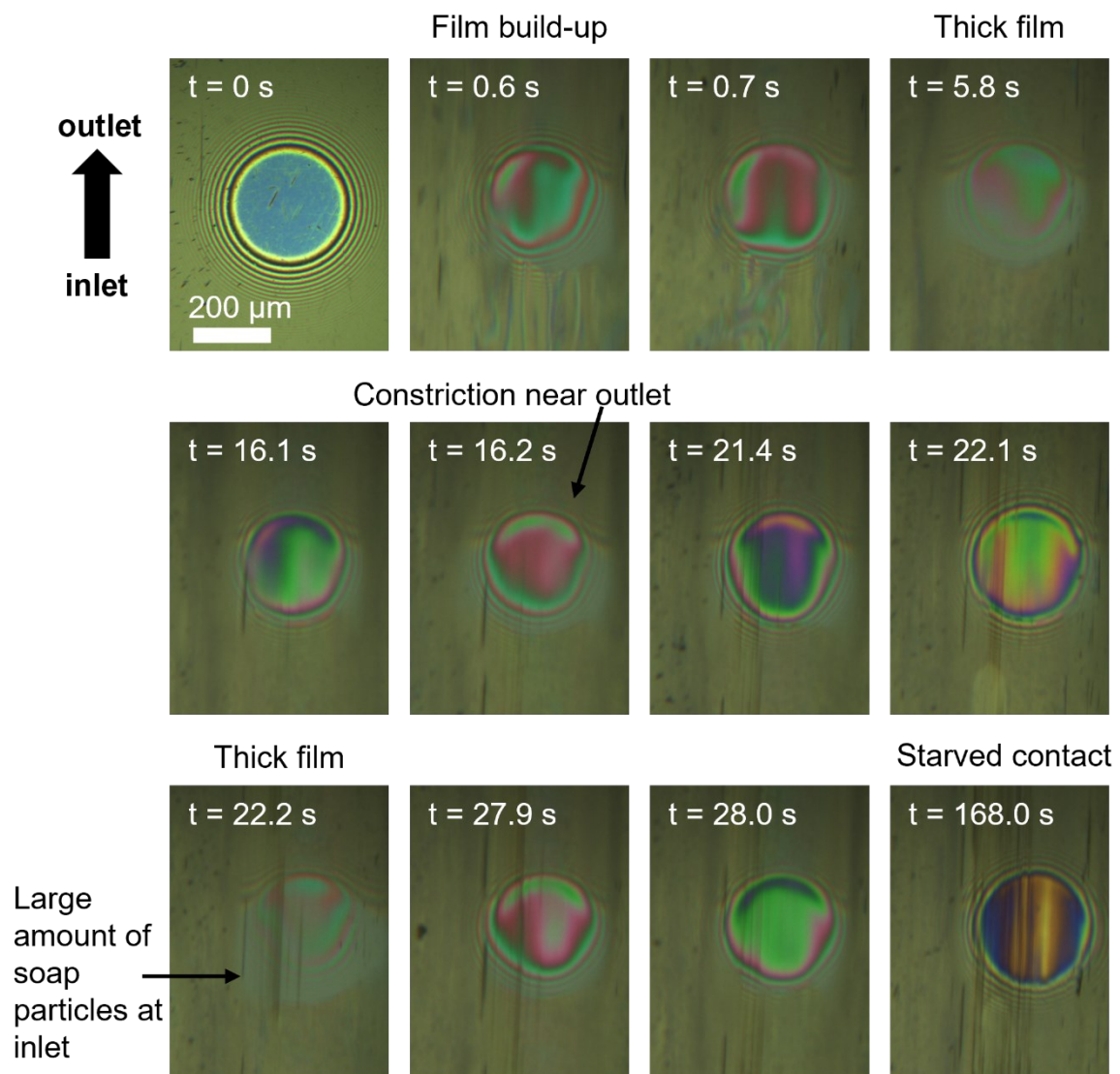


Figure 6-1: A series of images representing frames from a video of soap films formed in the contact between a steel ball and a glass disc on the EHD-HS rig (sodium soap, at 0.5 GPa, $SRR = -180\%$, $u_e = 1.0$ m/s, $u_s = 1.8$ m/s, $T = 40$ °C)

To investigate the soap film behaviour at speeds closer to real wire drawing speeds, the entrainment speed was increased to 10 m/s. As mentioned in chapter 4.1, the ETM tests showed very similar friction at all SRRs over 100%. Thus, to reduce damage of the glass disc at high speeds, the SRR was reduced to 100% in these high-speed tests.

Figure 6-2 shows frames taken from a 10 minutes test conducted at $u_e = 10.0$ m/s, $P_{max} = 0.5$ GPa, and $SRR = -100\%$. Similar to lower speeds (Figure 6-1), it can be seen that the produced films are very dynamic in time. Distinct features such as thicker and thinner films, starvation, and a constriction near the outlet are visible at different times, as indicated in the relevant images. At this high speed, the soap films at times assume shapes that are similar to conventional EHD oil film shapes with a flat central plateau and a constriction at the outlet, see for example image at $t = 65.5$ s in Figure 6-2. However, this EHL-like film does not persist and almost no film is recorded at other times in the same test, e.g. $t = 28.0$ s in Figure 6-2.

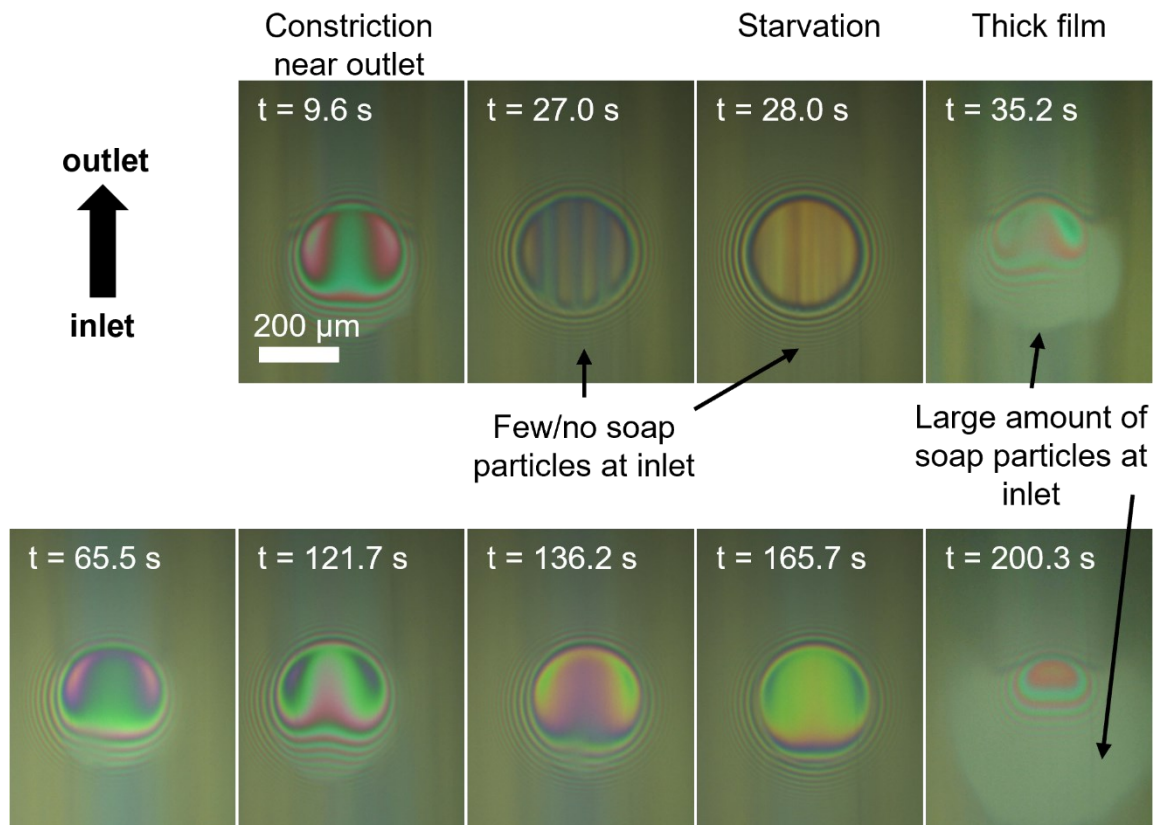


Figure 6-2: A series of images representing frames from a video of soap films formed in the contact between a steel ball and a glass disc EHD-HS rig (sodium soap, at 0.5 GPa, $SRR = -100\%$, $u_e = 10.0$ m/s, $u_s = 10.0$ m/s, $T = 40$ °C)

Further experiments were conducted at different SRRs and speeds. Figure 6-3 shows some of these results where the constriction at the outlet is apparent at six sets of high speed, high sliding conditions. An EHD-like horseshoe shape is evident in these images, too.

Together, these results indicate that the soap is able to form lubricating films in the ball-disc contact that at times assume features similar to those of conventional EHD oil films, especially at higher speeds, but at other times completely collapse.

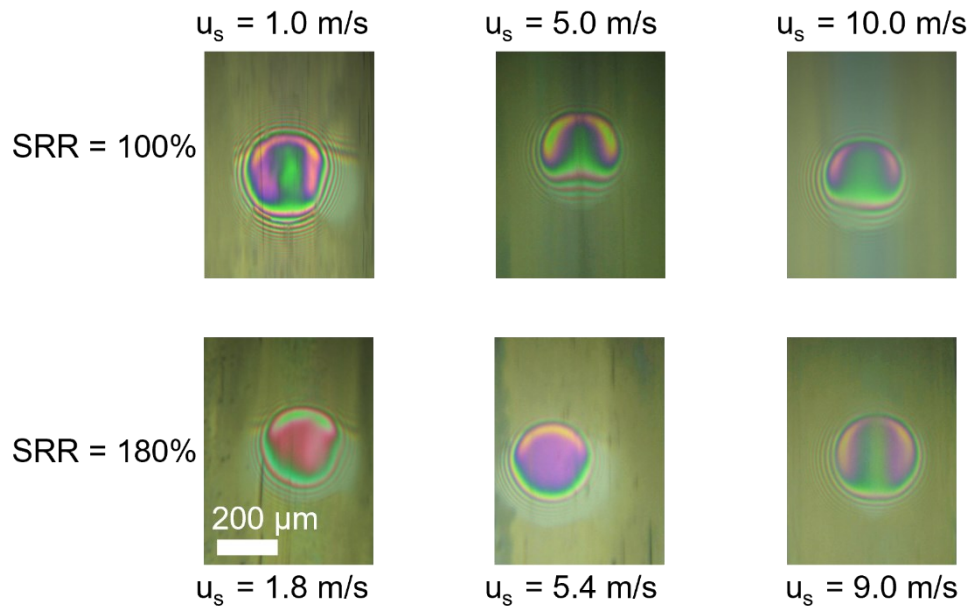


Figure 6-3: EHD-HS observation of sodium soap film at different conditions (as indicated) showing the dynamic nature of the film and EHD-like film shapes present at certain times ($P_{max} = 0.5$ GPa).

To study the effect of pressure on soap films, the glass disc was replaced with a sapphire one and pressure increased to ~ 1 GPa. Similar dynamic film features such as thicker and thinner films and starvation, were seen at this higher pressure as those already described above. Hence, Figure 6-4 shows only snapshots of the constriction near outlet at different speeds and SRRs. It must be noted that the sapphire disc used has a different coating than the glass disc and thus, the colours in the images with the sapphire disc are different to those with the glass disc.

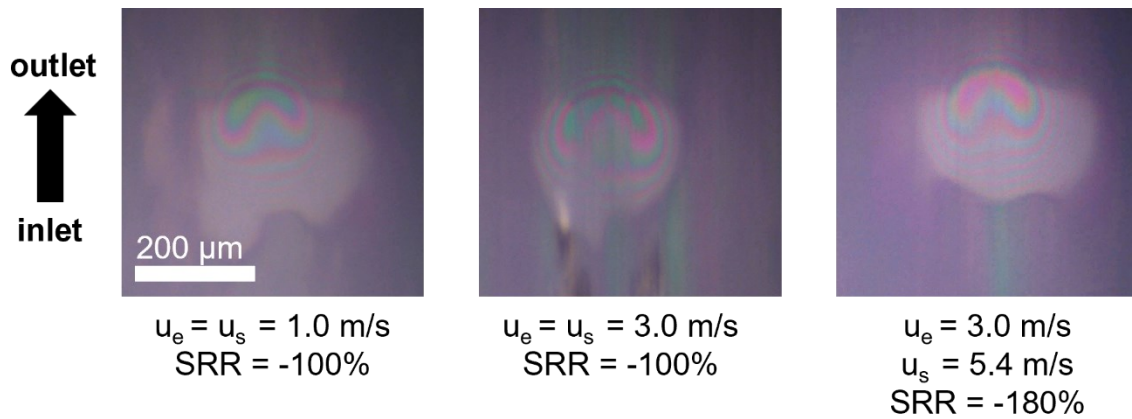


Figure 6-4: Snapshots of soap films in ball-disc contact on the EHD-HS rig using a sapphire disc (sodium soap, $P_{max} = 0.98 \text{ GPa}$, speeds and SRRs as indicated, all steady state conditions).

Taking a closer look at the results above, in particular in Figure 6-1 and Figure 6-2, the images show that the shape of the film in contact appears to be correlated with the amount of soap particles at the inlet (i.e. with soap supply to the contact). For example, when starvation was observed, e.g. $t = 27.0 \text{ s}$ in Figure 6-2, no soap particles are seen at the inlet. When more soap particles are present in the inlet, a more uniform film with an EHD-like constriction near the outlet can be seen, e.g. $t = 9.6 \text{ s}$ in Figure 6-2. When thick films are formed, a large amount of soap particles are apparent at the inlet, e.g. $t = 200.3 \text{ s}$ in Figure 6-2. These trends suggest that the film formation strongly depends on the entrainment and quantity of the soap particles into the contact.

6.2 Direct Observation of Soap Particles Passing Through the Contact

In view of the above observations and the apparent significance of soap particle entrainment into the contact, an attempt was made to directly observe individual soap particles entering and passing through the contact. For this, the EHD-HS pot was not filled with soap, but the ball was immersed in a bowl with soap before testing. Some particles would then adhere to the ball surface, after which the ball was mounted on the test rig. It was made sure that the initial contact was free of any soap particles so that the movement of 'single' particles entering the contact could be followed. To be

able to better follow the particles, the tests were conducted at very low speeds of 1.0 mm/s and a pressure of 0.42 GPa ($W = 10.0$ N).

The entrainment of a small, single particle in the contact is demonstrated in Figure 6-5. As soon as the particle enters the contact ($t = 1.0$ s and $t = 1.14$ s), the particle is squeezed and elongates ($t = 1.14$ s and $t = 1.28$ s). Upon exiting the contact, the deformed particle remains on the disc surface, forming a transparent film on the disc ($> t = 1.46$ s).

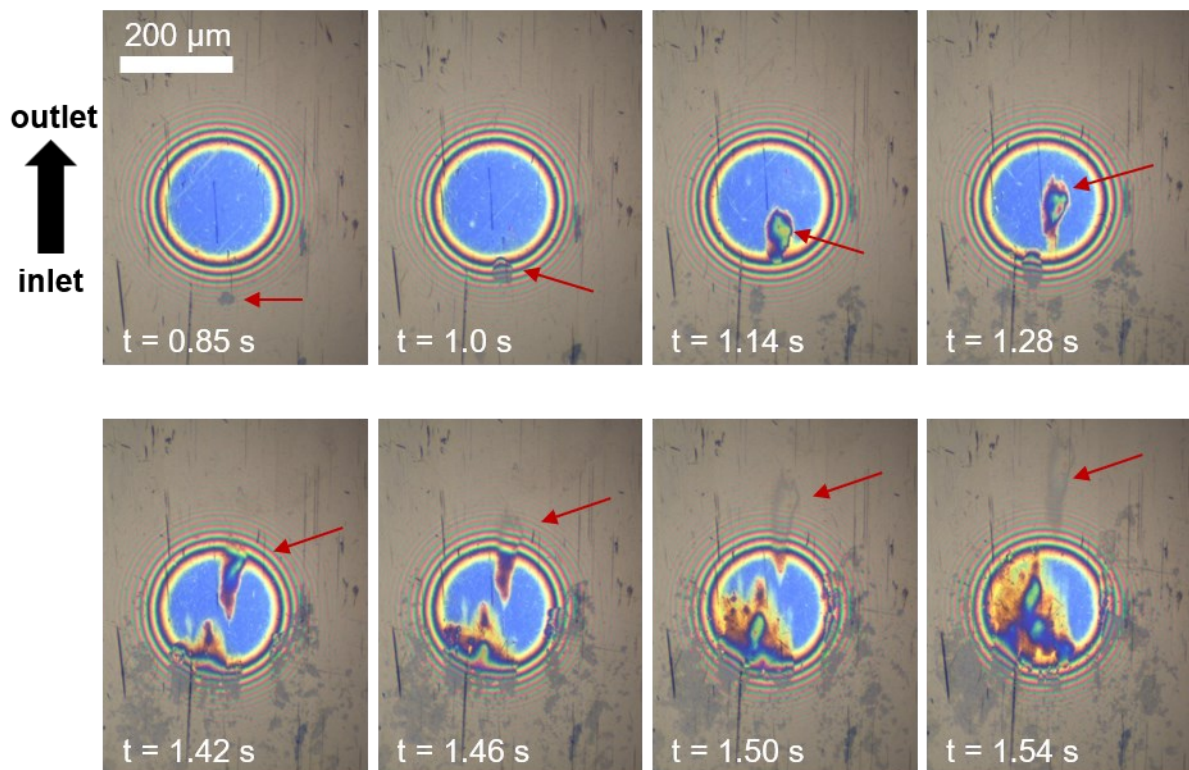


Figure 6-5: A series of images from a single EHD-HS test showing a soap particle entering, passing through and exiting the contact. The red arrows point to the particle at different times (see relevant text for more details). Sodium soap, $P_{max} = 0.42$ GPa, $u_e = 1.0$ mm/s, $SRR = -100\%$

A bigger sodium particle, or a conglomeration of particles, passing through the ball-disc contact is shown in Figure 6-6. It can be seen that the film in the contact ($t = 0.04$ s) is quite thick. At the outlet, the exiting particle seems to adhere to the disc surface and is elongated slightly ($t = 0.10$ s), but the shape of the particle remains unaffected. The developed ‘film’ on the disc surface after exiting the contact also seems to be very thick compared to the transparent film formed with the smaller particle in Figure 6-5 as it

seems to be less transparent.

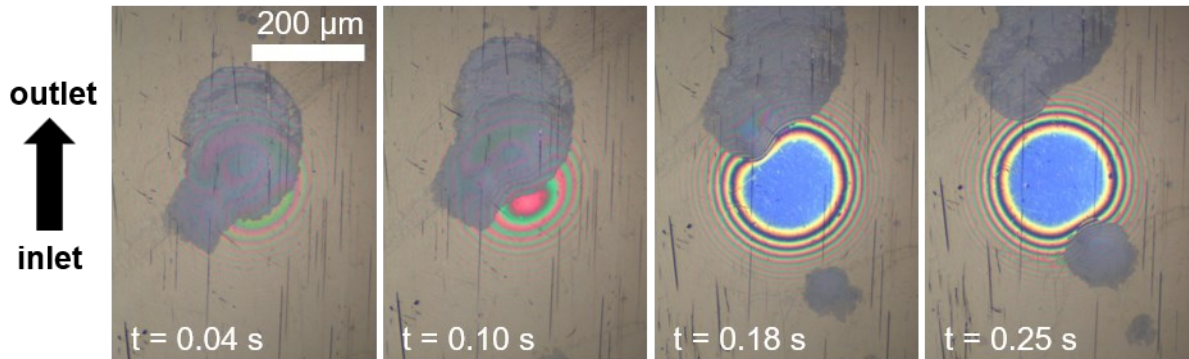


Figure 6-6: A series of images from a single EHD-HS test showing a bigger soap particle (or conglomeration of particles) entering, passing through and exiting the contact (sodium soap, $P_{max} = 0.42$ GPa, $u_e = 1.0$ mm/s, $SRR = -100\%$).

Figure 6-7 shows more and perhaps bigger particles entering the contact. For comparison, results for the two studied calcium soaps are also shown in this figure in parallel to the sodium soap ones. It is apparent in these images that similar films to those described above for sodium soap are also formed by the calcium soaps. As was seen in Figure 6-5, the particles form a transparent film on the disc surface once they exit the contact. Interestingly, for the low FA (35%) calcium soap, this transparent film seems to be narrower than the contact diameter. Taking a closer look at the film in contact, the film only forms half of the constriction near the outlet (e.g. as seen for the sodium soap), and only where this apparent thinner in-contact film (constriction near outlet) is seen, a transparent film is developed outside the contact ($t = 4.03$). Hence, this transparent film could indicate a thinner film compared to the part outside of this narrow 'track' which could suggest a thicker formed film. This also becomes more obvious when looking at the images for the medium FA content (55%) calcium soap. Here, a transparent film is not developed in the beginning ($t = 2.43$ s) but seems to only form afterwards (e.g. $t = 2.54$ s), again following a thinner film in the central region of the contact.

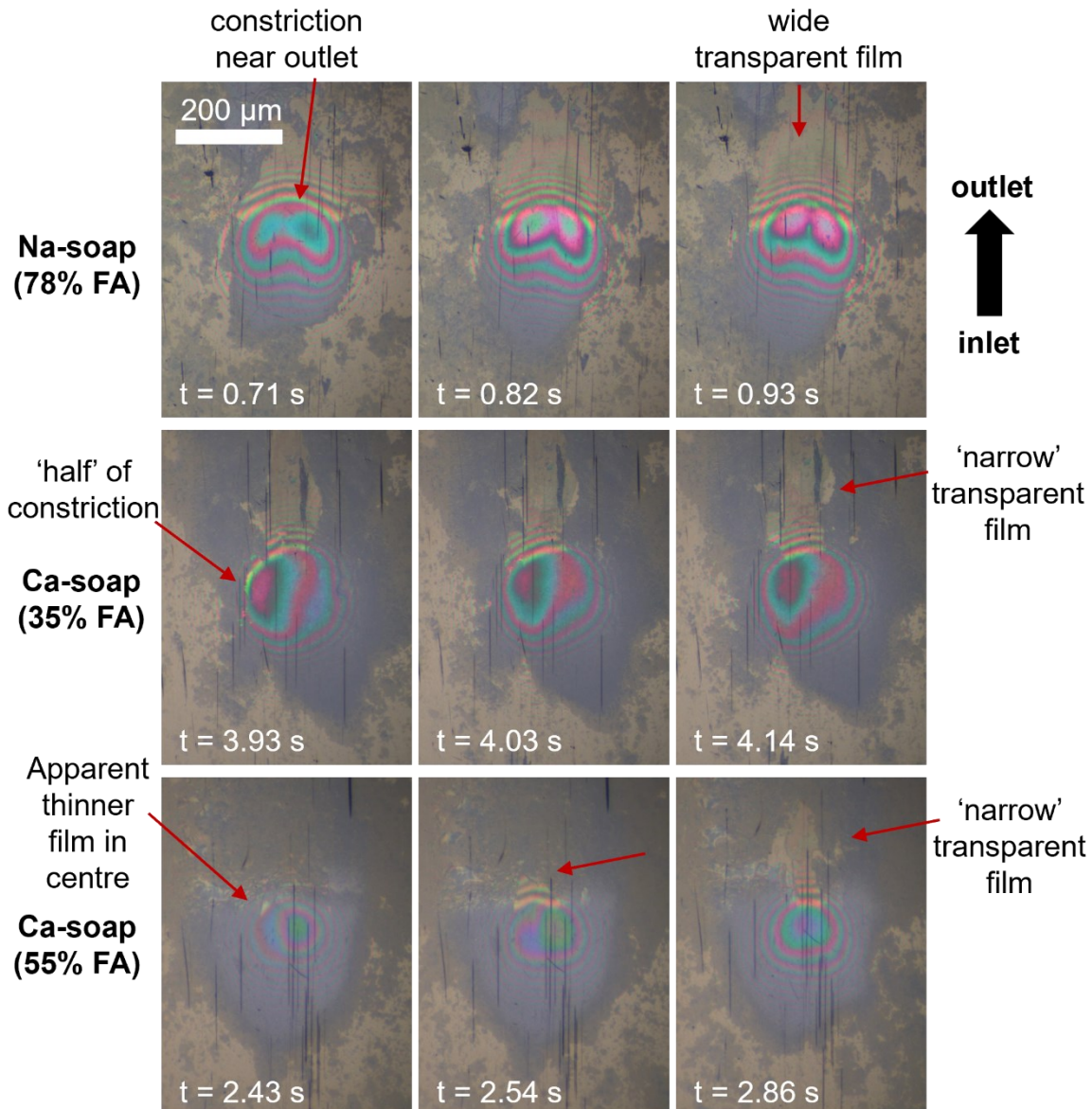


Figure 6-7: A series of images from three EHD-HS tests showing soap particles entering, passing through and exiting the contact (sodium and calcium soaps, $P_{max} = 0.42$ GPa, $u_e = 1.0$ mm/s, $SRR = -100\%$).

6.3 Simultaneous Observation of Film and Friction

The EHD-HS machine also has the ability to measure friction simultaneously with the film observations using a reaction torque sensor on the ball carrier cage, e.g. as shown by MacLaren and Kadiric (67). The method is not perfect as it still includes some parasitic torques (for example from the three supporting bearings on which the ball rests in the chamber although these are minimal in the present design), but it is much better

than simply using a torque meter. The EHD-HS was used to measure friction at the same time as observing the soap development in the contact in an attempt to correlate the two.

Figure 6-8 shows one example of EHD-HS friction measurements over a period of 10 minutes at constant conditions of 0.5 GPa, -100% SRR and an entrainment speed of 3.0 m/s. Snapshots of soap films in the contact at different times in the test are also shown in the figure. It is worth noting that the snapshots shown in this figure and friction measurements are not perfectly aligned as there is a latency between the friction data obtained and the film recordings. This latency is small and probably irrelevant for the current purposes of qualitative comparisons. Images from the highlighted regions where friction is more or less stable give good indications of changes in film behaviour. In the initial stages of the test (<100 s), friction is relatively high at ~ 0.08 . Recordings of the contact during the same period show that no significant soap film is present in the contact (as can be seen in the included example images). Towards the end of the test (>400 s) friction is very low at ~ 0.04 . During this time, a very thick film with some EHL-like features (flat central region and an apparent constriction near the outlet) can be seen in the corresponding images. In terms of actual friction measurements, it can be seen that measured EHD-HS friction is somewhat less stable in time than friction measured on the ETM. This is most likely due to the different set-up in the EHD where entrainment of soap particles is strongly dependent on the amount of soap particles adhering to the ball surface. In contrast, on the ETM set-up, the soap particles are being pushed back on the rubbing track by the scoop.

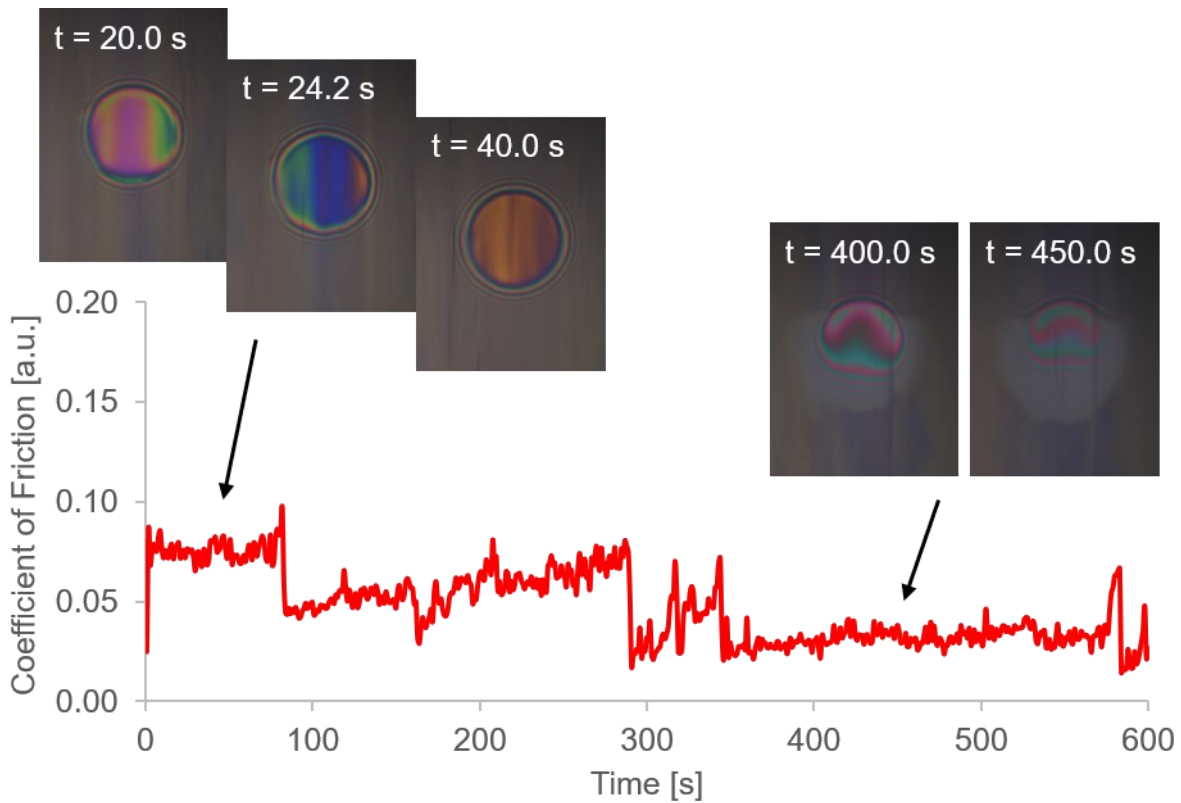


Figure 6-8: EHD-HS friction measurements over time at steady conditions and the corresponding images of the soap films obtained simultaneously (sodium soap, 0.5 GPa, $SRR = -100\%$, $u_e = 3.0$ m/s, $u_s = 3.0$ m/s).

Figure 6-9 shows further, several friction measurements conducted on the EHD-HS corresponding to film observations shown in section 6.1. Despite more instabilities throughout the test, the observed trends in Figure 6-9 again match those recorded on the ETM. In all cases, a short running-in period is seen where friction decreases. Furthermore, there are no significant differences between a SRR of 100 % and 180 %, again aligning with ETM friction results (chapter 4.1) and film observations above (chapter 6.1). In terms of the tested speed, it is clear that an increase in speed leads to a decrease in friction.

The similarities in the friction trends recorded on the ETM and EHD-HS, and the fact that both rigs record similar and very low values of steady-state soap friction of around 0.04 and below, provide further confidence in the presented results.

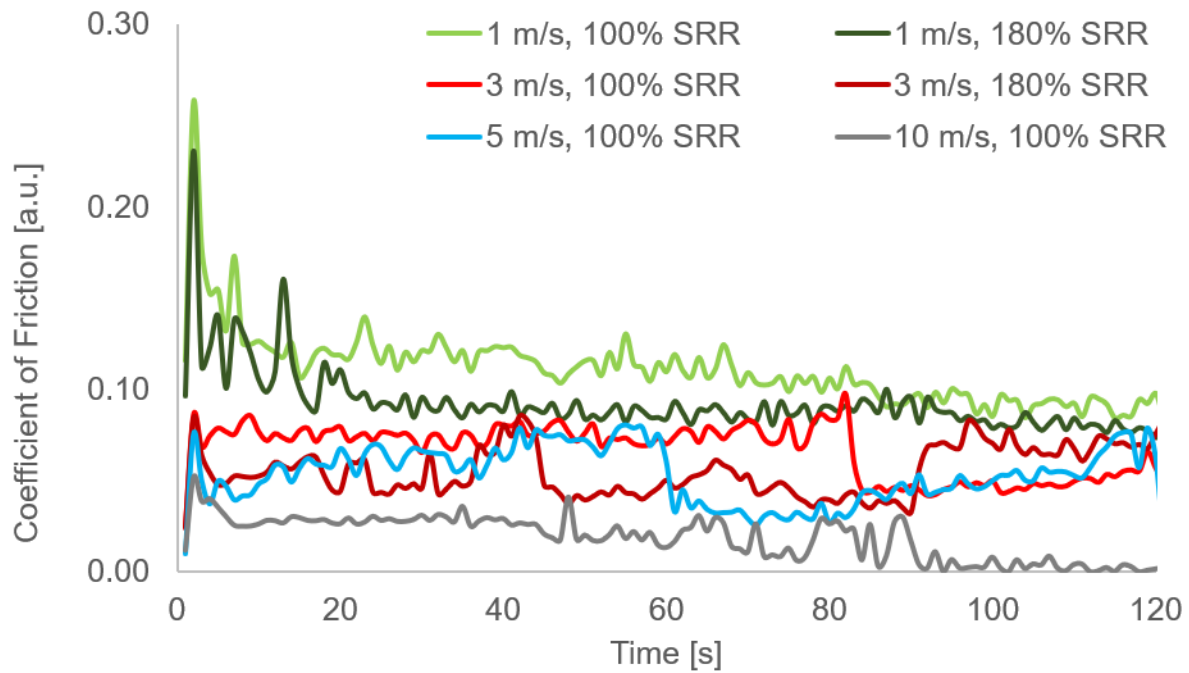


Figure 6-9: EHD-HS friction measurements with the sodium soap for a range of entrainment speeds and SRRs ($P_{max} = 0.5$ GPa), note: SRRs are negative.

6.4 Discussion of Soap Film Build-up

The formation of a constriction near the outlet at high shear and high speeds is quite interesting, in particular if compared to an EHD film of an oil. In fact, this constriction does resemble the characteristic ‘horse-shoe’ shape of a standard oil, e.g. (77–80). At first sight, this might suggest that soaps, at least intermittently, behave like a fluid following a phase transition as was also assumed in past literature, e.g. (46,52,53). From this perspective, it is interesting to study the influence of temperature on soaps’ physical properties, which is done in chapter 8.

Closer inspection of the entrainment of individual or few particles into the contact (section 6.2), give some indication about the lubrication mechanisms of soap powders. The soap particles seem to follow the behaviour of debris particles which are deformed and squeezed into shapes of platelets (81). The contact load is therefore high enough to deform these particles which then adhere to the surface of the specimens and form a soap film. This also applies for bigger particles in which case the formed film is quite thick. Given the large scale of grain sizes of soaps, the soap film varies significantly

throughout tests resulting in the dynamic features shown above. However, it is still not quite clear if the soaps also undergo a phase transition at the same time. The nature of those films is discussed in more detail in Chapter 9.2.

Above tests also show that the usually opaque soaps seemingly form an optically transparent lubricating film with interference fringes that can be observed in an optical machine. It would now be interesting to characterise the films in terms of thickness, but this is not yet possible. One major issue here is the lack of knowledge of refractive indices of the soap films. Additionally, post-film characterisation as well as EHD-HS observations show that soap films are very thick. With the current optical set-up, it is only possible to measure films of a few hundred nanometres and soap films appear to be thicker than this, at least locally. The variable nature of soap films in space and time also makes it difficult to determine an inlet thickness value that describes the films appropriately. More work has to be done to characterise the film thickness using the EHD-HS optical set-up but perhaps also other methods such as fluorescence for thick films.

The simultaneous observation of friction and film development allows to identify reasons for changes in friction. Lower friction was usually achieved when the contact was fully separated by a thick soap layer, whereas starvation or non-continuous films led to higher friction. Given the similarities in frictional trends between these two machines, this knowledge can therefore be transferred to the friction results from chapter 4.

Long-term friction tests (chapter 4.3) showed that friction is usually quite stable, especially for the studied sodium soap. A thick film separating ball and disc is most likely present here. After some time, when friction becomes unstable, the contact is probably starved similar to what was observed in Figure 6-8. Hence, the instabilities in above friction tests are presumably caused by the dynamics of the soap films.

7 Tests on Real Wire Drawing Machine

This chapter entails tests conducted on a real wire drawing machine. The intention at the start of this PhD was for the author to spend time at the sponsors R&D facilities to use their “Universal Wire Drawing Test Machine (UWT)” herself, but the outbreak of the Covid-19 pandemic prevented this from happening. Instead, the machine was run by the sponsor’s personnel to obtain the results in section 7.1 immediately below, but the author was involved in the analysis of results presented in section 7.1.

7.1 Results of UWT Drawing Tests

The experimental tests presented in this subchapter were performed on the UWT at Bekaert’s Technical Centre in Belgium by Stijn de Pauw and Hendrik Van Hoecke of Bekaert. The author contributed to the analysis of the results. The UWT set-up is described in section 3.8.

Figure 7-1 shows the energy consumption per mass of wire drawn measured on the UWT as a function of drawing speed for a set of drawing die angles during experiments with sodium-based soaps on the UWT. Since the mass flow increases with drawing speed, the energy consumption per mass was used as value for comparability. The highest energy consumption at higher speeds (≥ 0.42 m/s) was reached by the largest two die angles of 30° and 35° but the lowest die angle of 6° does not lead to the lowest energy consumption. Lowest energy consumption is achieved with a die angle of 9° . With an increase in die angle energy consumption increases. It can be seen that energy consumption per mass unit generally decreases with increasing average drawing speed. For $2\alpha = 9^\circ$, an energy usage reduction of 20.5% is achieved by increasing drawing speed from 0.08 m/s to 4.17 m/s (decreasing from around 78 kJ/kg at 0.08 m/s to about 62 kJ/kg at 4.17 m/s). The energy reduction with speed occurs for all tested die angles, except for the largest angle of 35° .

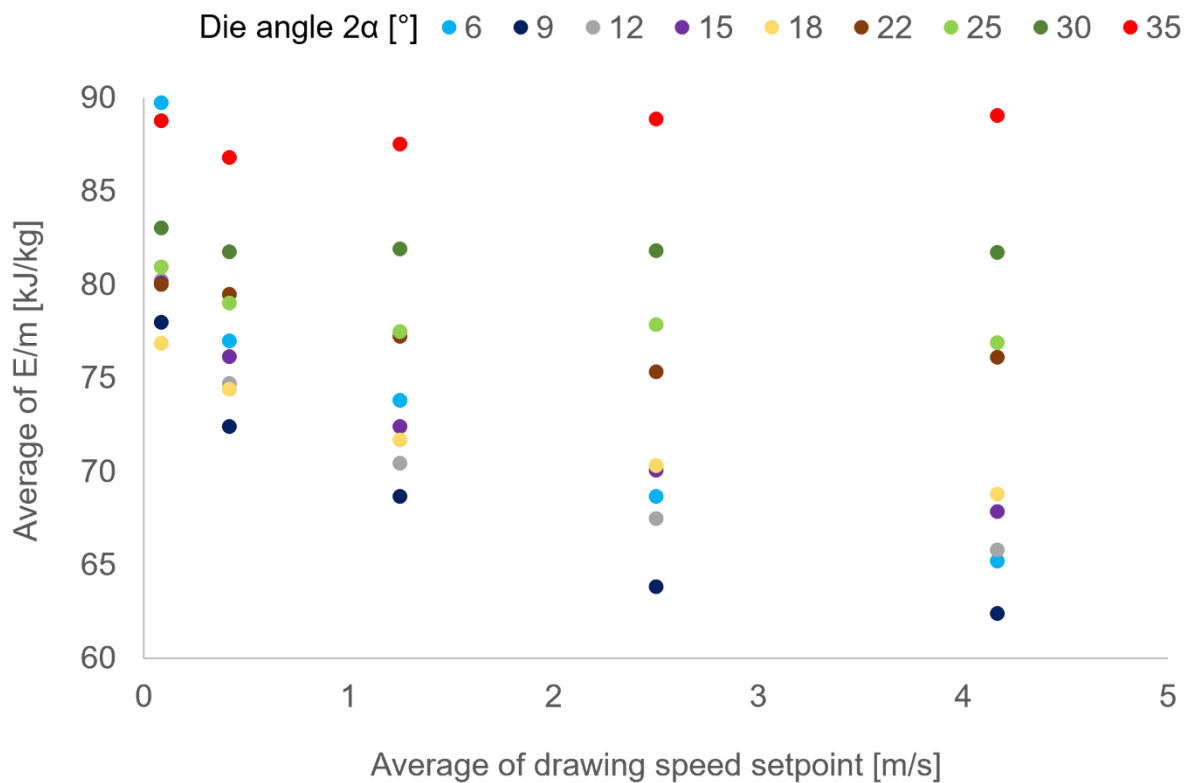


Figure 7-1: Measured energy consumption during dry wire drawing on the Universal Wire Drawing Machine (UWT) at Bekaert as a function of drawing speed and die angle with sodium-based soaps.

7.2 Wire Drawing Samples

Large parts of this subchapter were published by the author in the open access paper (41).

To assess whether the residual soap films observed after ETM tests are representative of those that may be found on real wires after drawing, a number of real wire samples of different diameters were also analysed. All analysed wires were drawn using the same sodium soap as used in the lab tests above and were taken from four selected, inconsecutive drawing dies from the same drawing machine. Immediately before entering the drawing die, the wire passes through a soap box so that the soap layer on the wire is always replenished. The original wire diameter in this case was 3.15 mm. Figure 7-2 shows confocal images and the corresponding topographies of the surfaces of real wire samples with different diameters. The smaller the wire diameter, the more drawing passes the wire has undergone. Some residual, inhomogeneous soap layers

can be seen on the wires. These soap layers are quite thick up to a few microns. With decreasing wire diameter (increasing number of drawing passes), less soap residuals are found on the wire surface, see for example the images for d_1 of 1.49 mm and 0.89 mm vs. those for d_1 of 2.46 mm and 1.90 mm. The thicker films at higher diameters also seem to be more lumpy.

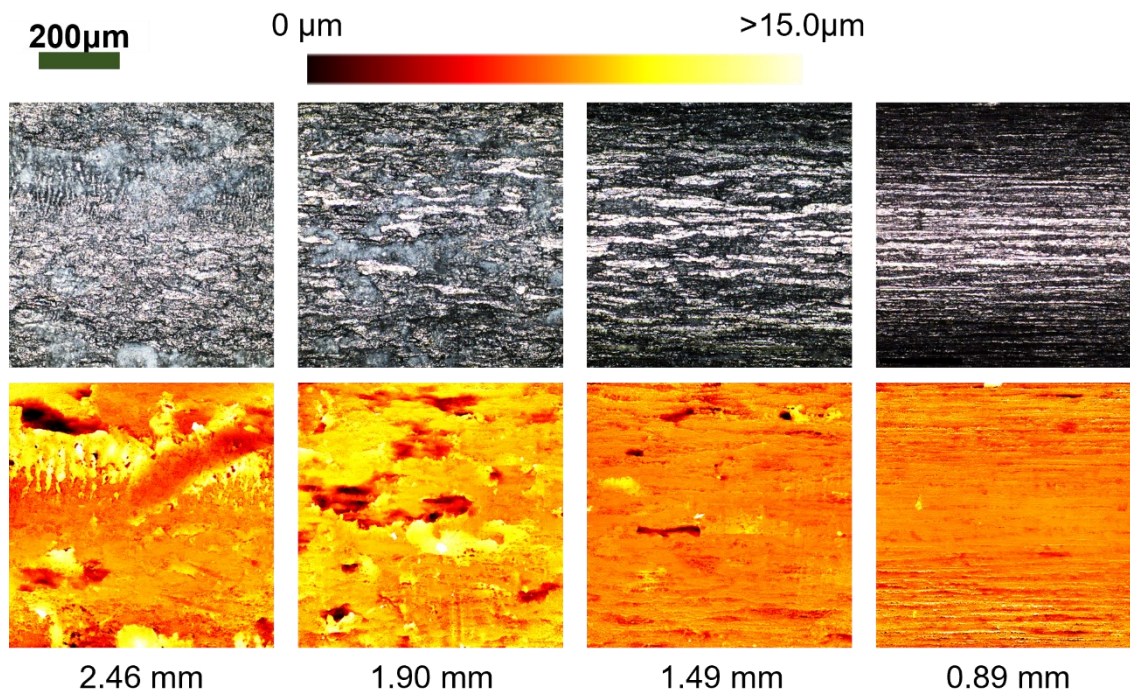


Figure 7-2: Example confocal images (top) and corresponding surface roughness profiles (bottom) of a set of real drawn wires with different diameters (diameters specified in bottom row).

The surfaces of the same wires were also analysed on the SEM-EDX. Figure 7-3 shows the weight percentage of sodium content on these real wire samples with different diameter. Sodium was detected on all wire samples. However, with decreasing wire diameter (increasing number of passes), the sodium content on the wires decreases, suggesting a thinner film or less coverage on the wires.

So, surface of both ETM discs post test and the real drawn wires show the presence of residual sodium films, again offering reassurance that the lab tests are capturing the relevant mechanisms taking place in real wire drawing. However, the absolute sodium content on the rubbing track of the ETM discs is much lower than that recorded on any of the four real wires analysed here. There could be multiple reasons for the

discrepancy in absolute Na content between ETM disc and real wires. For example, the ETM disc undergoes multiple revolutions, so it is over-rolled by the ball multiple times in a test; given the trend on real wires for decreasing Na with increasing number of passes, a relatively thin residual film on ETM discs may perhaps be expected.

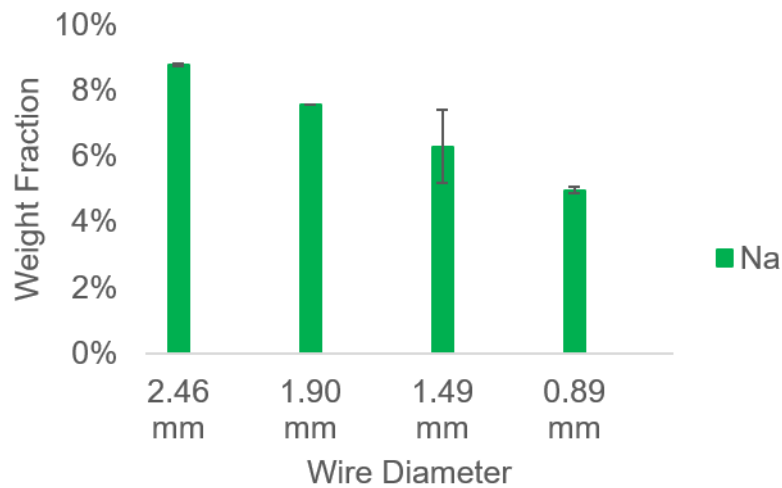


Figure 7-3: wt. % Na measured by SEM-EDX on the surface of a selection of real wire samples with different diameters (landing energy = 20 keV).

7.3 Discussion

The wire drawing experiments on the UWT show that the die angle is a crucial element in reducing energy consumption. Low die angles generally led to lower energy consumption. Similarly, a decrease in die angle was proven to reduce drawing stresses, e.g. (9,36,82). In above wire drawing tests, a die angle of 9° compared to a die angle of 35° at 4.17 m/s could lead to energy savings of around 30%. Thus, optimising the die angle could lead to major energy reductions in the real application.

The trends observed on the real wire drawing machine seem to be broadly in line with the conducted ETM friction tests. For example, above measurements on the UWT showed that an increase in drawing speed led to a decrease in energy consumption. A decrease in energy consumption might be directly linked back to a decrease in drawing stress as was also presented for example in (9). This aligns well with the observations in chapter 4.2 where friction decreases with increasing speed. Moreover, for

bigger drawing die angles, energy consumption first decreases up to a speed of 1.25 m/s after which it increases slightly.

Studied wire samples also show that a residual film is prominent on the wire surfaces. Wires with a bigger diameter (early stage in wire drawing process) indicated some thicker films and more coverage which aligns well with SEM-EDX analysis. With decreasing wire diameter and increasing drawing pass, the soap layer coverage reduces. The reasons for this have not been studied in this work but a possible cause could be wear of the borax coating on the wires which in return might lead to reduced soap pick-up.

The residual sodium soap films on the wires are much more inhomogeneous compared to the residual soap film on the rubbing track of the ETM discs. One reason for this could be the different roughness of the specimens. The used ETM discs in this work were very smooth for better control of testing parameters and different roughness parameters were not studied. This should be done in future work to better compare produced films.

In summary, it was shown that ETM friction trends align well with energy consumption measurements on a wire drawing machine. One major advantage of the ETM is to measure friction directly which is not possible on the real wire drawing machine. Furthermore, investigations of wire samples gave evidence of residual soap layers relatively similar to soap films generated on ETM specimens. Thus, the ETM experiments appear to be able to capture at least some of the mechanisms at play in real wire drawing, proving further reassurance that the lab results shown here can be used to better understand the tribology taking place in the real die-wire contact.

8 Temperature Development in Contact

The low friction coefficients achieved with soaps at high pressures and high speeds, as well as the observation that at higher speeds soap films at least intermittently resemble the characteristic 'horse-shoe' shape of an EHD film with a fluid, may suggest that soaps might be behaving like a fluid or at least a very compliant gel-like solid at these conditions. Under these high-speed (high shear rate), high-pressure conditions, the contact temperature increase is significant. Therefore, this raises the question if the soaps undergo a phase-transition or at least a significant softening due to these elevated temperatures, which then allows them to form effective, low-friction lubricating films. To investigate this, firstly, the in-contact flash temperature rises are calculated for above tests, and secondly, the temperature-dependent phase-transitions of studied soaps is characterised using differential scanning calorimetry (DSC). Parts of this chapter have been reported by the author in the open access paper (41).

8.1 Flash Temperature Rise in Contact

High pressures and high sliding speeds lead to frictional heating which affects underlying tribological conditions (83). When two rubbing surfaces are moving with the velocities u_a and u_b , and u_a is greater than u_b , the rate of heat ($\dot{q}$) generated at the contact is given by:

$$\dot{q} = F|u_a - u_b| = W \cdot COF \cdot |u_a - u_b| \quad (8.1)$$

where F is the frictional force and W is the applied normal load.

Within the real area of contact, the heat flux can then be expressed as follows:

$$\dot{Q} = COF \cdot P|u_a - u_b| = \frac{\dot{q}}{A_c} \quad (8.2)$$

where P is the contact pressure and A_c is the contact area.

This frictional heating leads to a flash temperature rise in the contact and also the bulk temperature rise of the rubbing bodies, including the temperature in the contact where

the soaps are entrained. These temperature rises have a strong effect on the physical properties of the lubricant, and therefore, directly impact the lubrication process (84). At very high temperatures, this can also lead to changes of the physical properties of the rubbing materials and even melting of the bodies (84).

To better understand the soap lubrication behaviour, it is therefore crucial to know the maximum temperature of the contact of two rubbing surfaces due to frictional heating in the present tests. It is assumed that all the frictional heat is conducted into the two solids, and none is convected away by the soap. The total contact temperature T_c was calculated as follows:

$$T_c = T_b + \Delta T_f \quad (8.3)$$

where T_b is the bulk temperature of a body (away from the contact). The maximum temperature increase at the contact surface is only of short duration and is referred to as the flash temperature rise ΔT_f .

To predict contact temperatures in a rough surface contact accurately, a numerical approach is required. However, there are several analytical expressions that can be used to estimate the flash temperature rise. These have different forms depending on the contact shape and the heat source distribution.

The flash temperature rise of a homogeneous, semi-infinite body can be identified by integrating the distributed heat flux input over the area of contact (85). ETM ball-on-disc contact has a semi-elliptical heat distribution with the maximum heat in the centre but for simplification the flash temperature rises here are estimated with a mean heat flux everywhere. Thus, for a circular heat source with a uniform heat flux distribution, Kennedy (84) provides a simple expression for the maximum flash temperature:

$$\Delta T_f = \frac{2\dot{q}a}{k\sqrt{\pi(1.273 + Pe)}} \quad (8.4)$$

where Pe is the Peclet number which measures how quickly heat is conducted into solids compared to how quickly the contact is transverses; it includes the velocity u of the moving body and is defined as (84):

$$Pe = \frac{ua\rho C}{2k} \quad (8.5)$$

In order to determine the temperature rise in a 'single' body, it is necessary to determine the heat partition factor, B , which gives information about the fraction of heat (B) going into one contacting body and the fraction of heat ($1-B$) going into the other (84). This is usually calculated by equating the mean temperatures of the two contacting surfaces and can be calculated as follows (86):

$$B = \frac{\frac{1}{k_b} \left(\frac{\chi_b a}{u_b} \right)^{0.5}}{\frac{1}{k_a} \left(\frac{\chi_a a}{u_a} \right)^{0.5} + \frac{1}{k_b} \left(\frac{\chi_b a}{u_b} \right)^{0.5}} \quad (8.6)$$

$$\chi = \frac{k}{\rho C} \quad (8.7)$$

where χ is the thermal diffusivity and describes the heat transfer within a material (86).

Most of the heat enters the faster body because it adds more new material per unit time to the contact (84). Once the heat partition into each body is worked out, the equations (8.4) and (8.5) can be used with the properties of either of the bodies to get the flash temperature rise.

The thermal properties of the materials used in this work as needed in these calculations are stated in Table 12. Based on the materials used the flash temperature rises could be calculated for above test conditions in the ETM and EHD (see Table 2 and Table 5).

Table 12: Thermal properties of materials used in ETM and EHD tests.

	Thermal Conductivity, k [W/m·K]	Density, ρ [kg/m³]	Specific Heat Capacity, C [J/kg·K]
C15E steel ETM discs (87)	25.0	7,700	460
WC ETM balls (88,89)	58.0	15,565	238
AISI 52100 steel EHD ball (90)	46.6	7,810	475
Glass EHD disc (91,92)	1.1	3,000	805
Sapphire EHD disc (93)	37.3	3,970	699

Figure 8-1 shows the calculated flash temperatures as a function of entrainment speed and sliding speeds at 180% SRR and different pressures tested on the ETM. An average friction coefficient of 0.03 and a bulk temperature of 40°C were assumed. The maximum flash temperature rise is estimated to be around 165°C at a pressure of 2.0 GPa, 215°C at 2.5 GPa, and 270°C at 3 GPa at the highest sliding speed employed in these tests of 3.6 m/s.

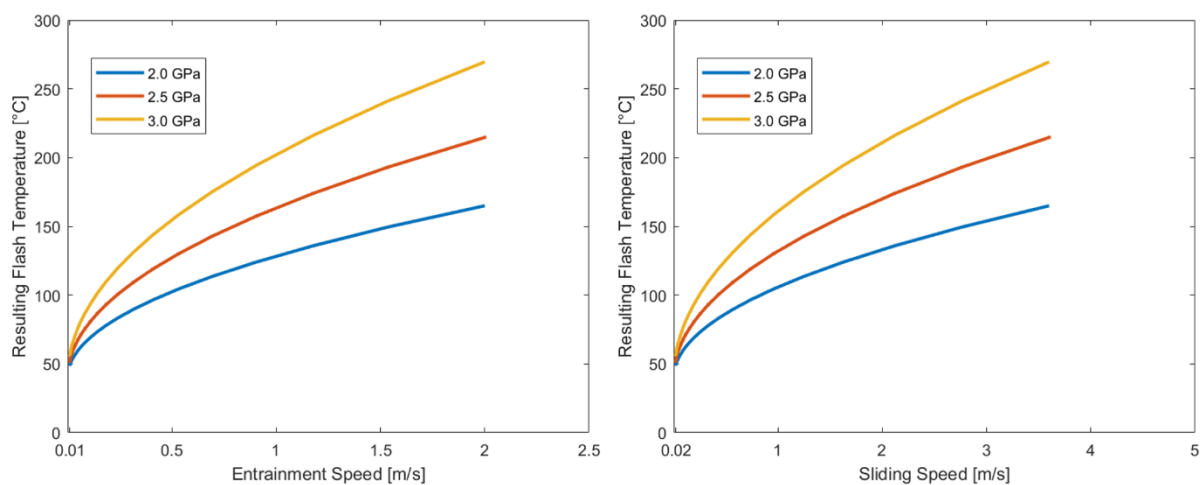


Figure 8-1: Estimated flash temperature rises in WC ball on steel disc contact under ETM test conditions ($SRR = 180\%$, $T_{bulk} = 40^\circ\text{C}$).

For the test conditions on the EHD-HS, the flash temperature rises, shown in Figure 8-2, are much lower since lower pressures were used. Again, a constant COF of 0.03 was assumed in the estimations. Since the EHD-HS tests were done at room temperature, a bulk temperature of 25°C was used in the calculations. The maximum flash temperature rise at highest entrainment speed of 5 m/s ($u_s = 9.0$ m/s) was estimated to be around 68°C and 83°C for the glass ($P_{max} = 0.5$ GPa) and sapphire ($P_{max} \sim 1.0$ GPa) disc respectively.

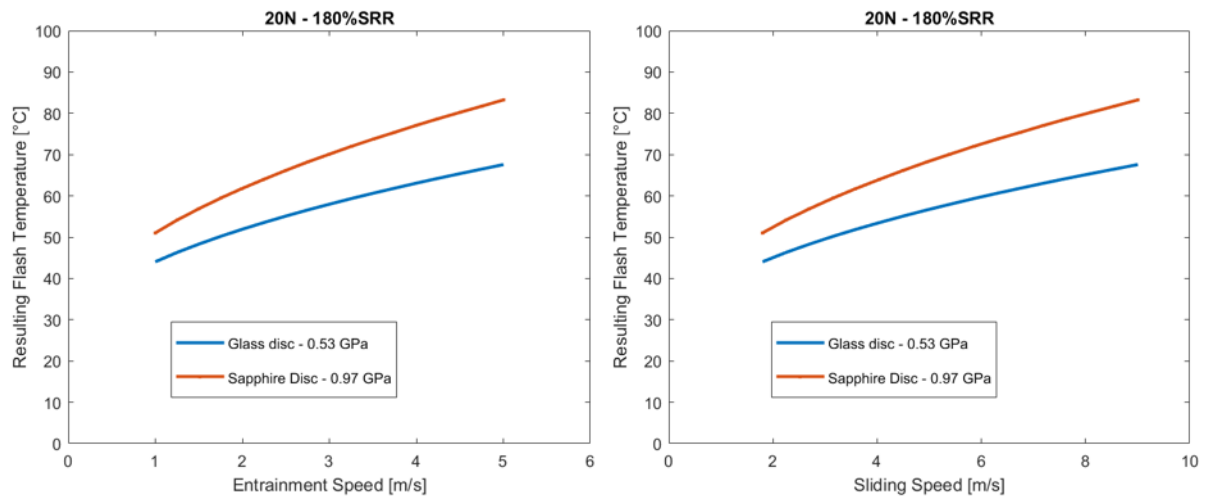


Figure 8-2: Estimated flash temperatures in steel ball on glass disc contact in the EHD-HS tests ($SRR = 180\%$, $T_{bulk} = 25^\circ\text{C}$).

8.2 Effect of Temperature on Soaps

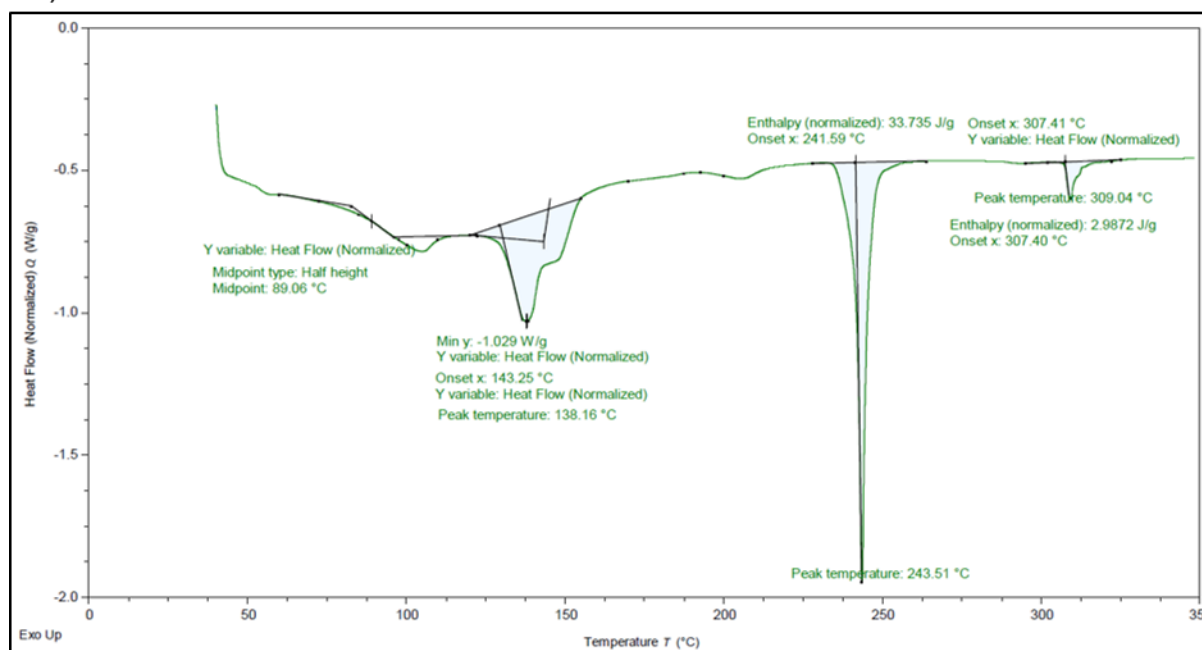
To investigate the effect of elevated temperatures on possible soap phase transitions, differential scanning calorimetry (DSC) measurements were conducted. DSC tests were only done for the two extremes of the studied soaps, namely the high fatty acid content sodium soap (78% FA) and the low fatty acid content calcium soap (35% FA).

Figure 8-3a shows typical DSC graphs with temperature increasing up to 350°C obtained for the studied sodium soap. Three repeats were done and shown graphs are typical results. Results showed a sharp endothermic peak at around 240°C. Closer inspection of the thermogravimetric analysis (TGA) in Figure 8-3b also showed no significant weight loss at this temperature. Hence, this peak indicates the melting temperature for this sodium soap. On the DSC-TGA graph (Figure 8-3b) a peak of the same order with minimal weight loss is detected at a slightly higher temperature of around

250°C on average. Estimated glass transition temperatures varied quite significantly between three repeat tests, as well as between DSC and DSC-TGA measurements, but seem to lay between 70°C and 140°C. At temperatures above 430°C, a huge mass loss is seen which implies that the melted soap evaporated.

DSC and DSC-TGA measurements conducted with the low FA (35%) calcium soap are shown in Figure 8-4. Three repeats were done and shown graphs are typical results. Results showed a sharp endothermic peak at 88°C (Figure 8-4a); these were much lower in magnitude compared to the sodium soap. In fact, in the DSC-TGA graph (Figure 8-4b) this peak is very small and could perhaps indicate the glass transition temperature. A strong endothermic peak is observed at temperatures between 440°C and 470°C (Figure 8-4) but an extensive weight loss is detected at the same time. This could suggest that no melting is occurring with the calcium soap, but the powder transitions directly from the solid to the gas state.

a) DSC



b) DSC-TGA

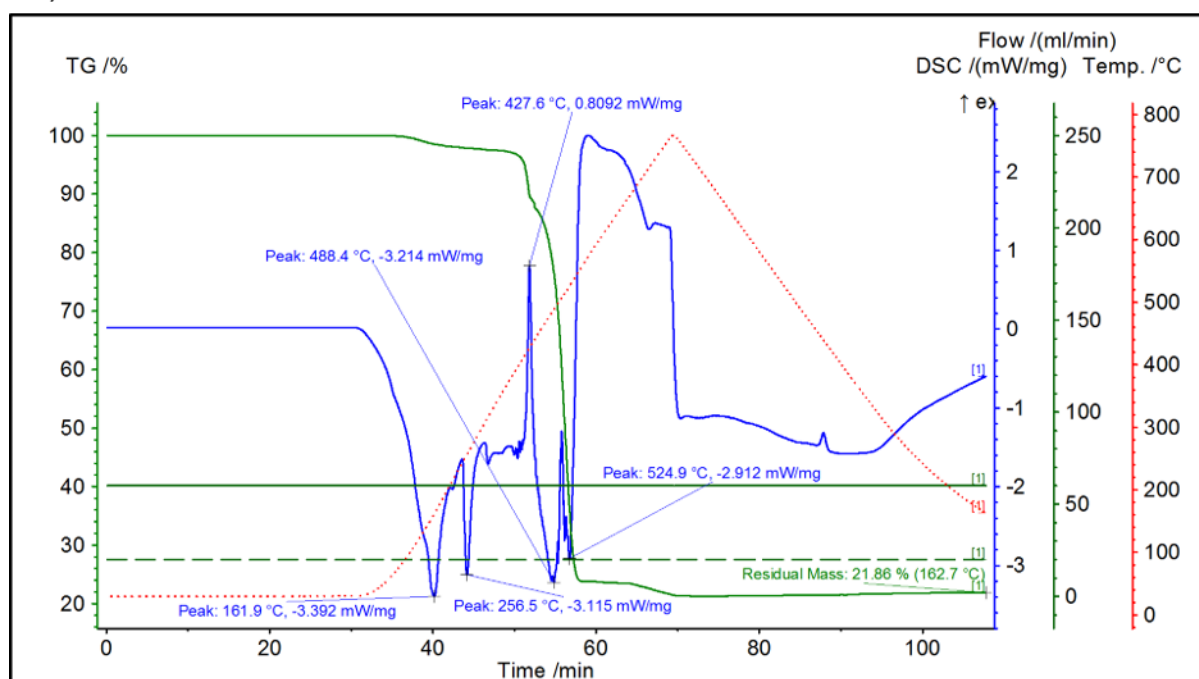
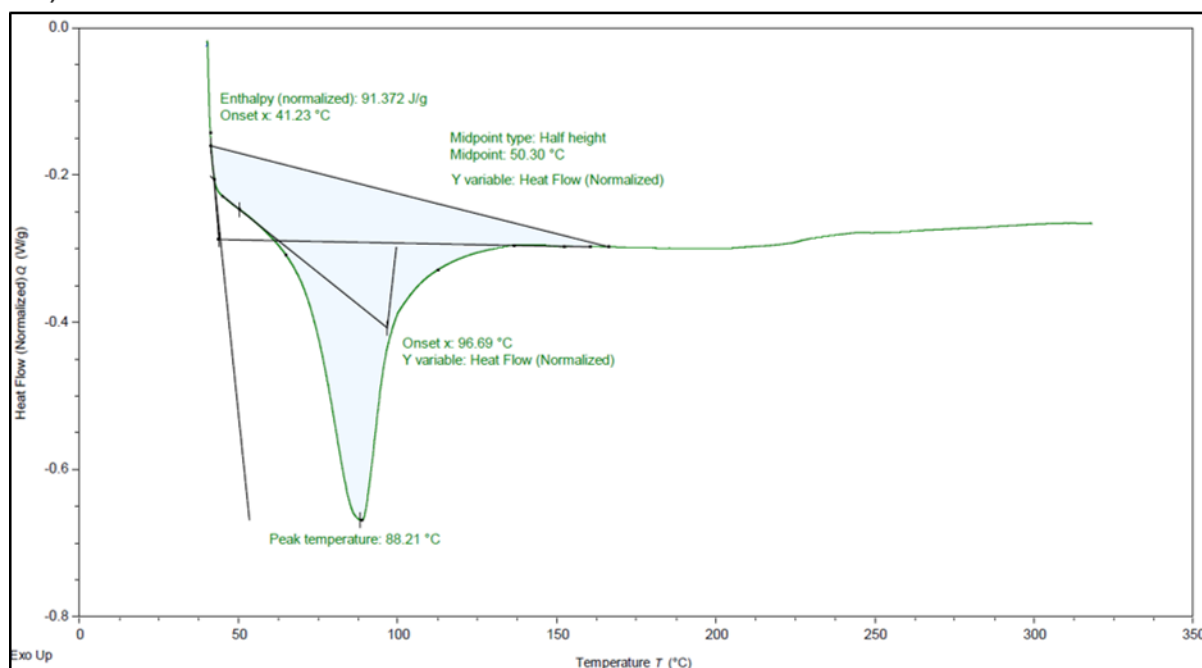


Figure 8-3: a) Typical Differential Scanning Calorimetry (DSC) measurements up to 350°C obtained for the sodium soap (78% FA), b) DSC and Thermogravimetric Analysis (TGA) up to 800°C.

a) DSC



b) DSC-TGA

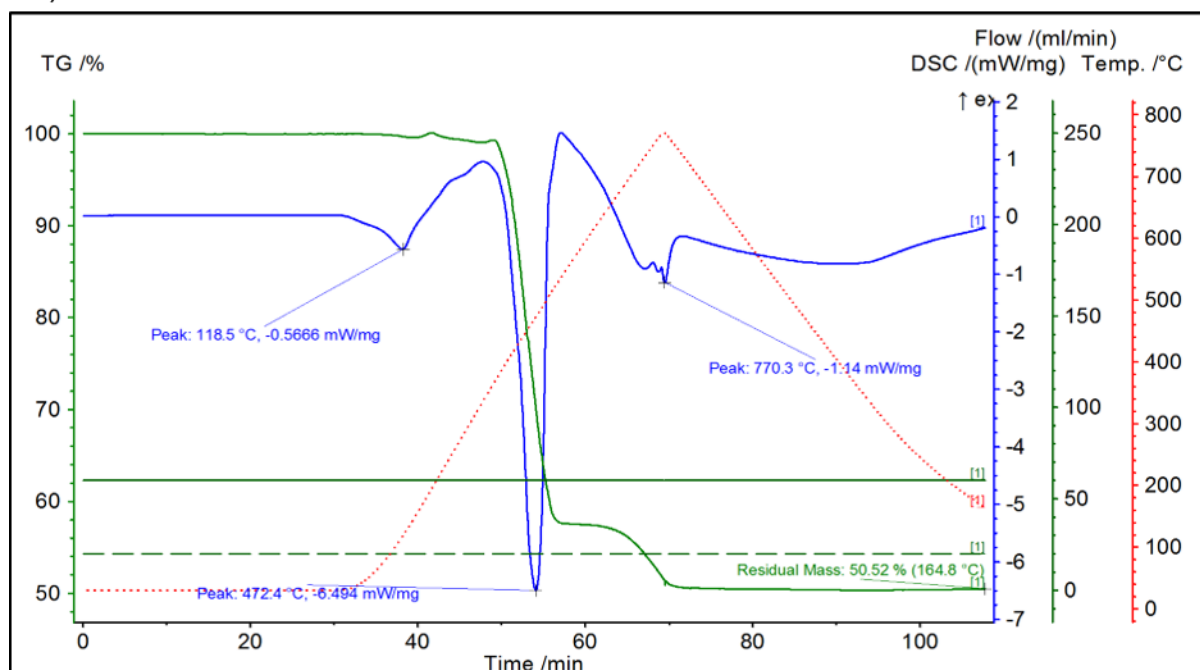


Figure 8-4: a) Typical DSC measurements up to 320°C obtained for the calcium soap (35% FA), b) DSC and TGA up to 800°C; note different scales between measurements.

8.3 Discussion

The flash temperature rises under ETM test conditions are estimated to be around 55°C at low sliding speeds ($u_s = 0.02$ m/s), and in the order of 200°C at high sliding speeds ($u_s > 1.5$ m/s). Friction results under these conditions (Figure 4-2) showed that friction is much higher at lower speeds, when flash temperature rises are also relatively low. With increasing speed, friction decreases which could be caused by the increase in in-contact temperature. Past literature suggests that a phase transition is seen for soaps at elevated temperatures above 200°C (51–53). It is also assumed that wire drawing soaps may melt at these temperatures (41).

Differential scanning calorimetry measurements with the sodium soap showed presence of significant peaks at temperatures of around 240°C to 250°C which most likely indicate soap melting. Past work on the physical properties and structures of metal soaps, see for example Vold and co-workers (94,95), Vold (96) and McBain *et al.* (97), also identified the transition of anhydrous sodium soaps from a subwaxy to waxy to liquid state with an increase in temperature. Therefore, it is entirely plausible that the low soap friction with the sodium soap observed at high pressure and high sliding speed conditions is caused by the powdered sodium soap transitioning to a fluid or wax-like state which may then lead to improved lubrication and perhaps a formation of a hydrodynamic film.

For the calcium soap, the DSC measurements did suggest some kind of reaction at high temperatures of 440°C and higher but seem to imply a sublimation process rather than melting. This aligns well with reports by Vold *et al.* (98) showing that calcium stearates melt with decomposition at high temperatures of 350°C. With closer look at the confocal images post friction tests (chapter 5.1), it might also seem that the produced calcium soap film is rather patchy at times, again indicating that no melting has occurred.

Since the estimated in-contact temperatures reached in the ETM tests at high sliding speed and pressure are in the range of 200°C – 270°C, it is unlikely that the calcium soaps melted. However, it has been reported that drawing forces with calcium soaps decrease at high temperatures between 100°C and 300°C (99). In addition, the tests on the EHD-HS (Chapter 6) showed that produced films resemble the horseshoe

shape of an EHD fluid or gel at least intermittently. Calculations done for the EHD-HS test conditions showed much lower maximum flash temperature rises, well below 100°C, but a constriction near the outlet still appeared in those tests. The estimated temperatures lower than the potential ‘melting’ point of the calcium soaps, the reported decrease in drawing force with calcium soaps at these temperatures (99), and the appearance of films resembling the horseshoe shape, raises the question if transitioning into a fluid state is needed to produce those films with low shear stresses. The reached in-contact temperatures in the EHD-HS tests are more in the order of the glass transition temperature of the calcium soap. Hence, this glassy, rather than fluid, state could be the reason for the low friction values obtained with powdered calcium soaps.

9 General Discussion

Each set of results presented in earlier chapters was accompanied by its own discussion. Here, some more general aspects of the present research are discussed. Some limitations of the experimental approach used in this research will be reviewed at the end of this chapter.

9.1 Soap Friction

The ETM ball-on-disc lab tests performed in this work showed that dry wire drawing soaps are able to provide very low friction coefficients; at high sliding speeds and high pressures, the measured COFs were as low as 0.03 for the high fatty acid (78% FA) sodium-based and the medium fatty acid (55% FA) calcium-based soaps, and around 0.04 for the low fatty acid (35% FA) calcium soap. These values are comparable to friction coefficients with typical oils under similar conditions. Sodium soap achieving the lowest friction coefficients, in particular at high speeds, aligns well with industrial wire drawing practice where sodium soap is the preferred soap and calcium soaps are usually employed when the sodium soap performance becomes inadequate⁴.

Different parameters such as sliding speed and pressure have a significant effect on friction. An increase in both, sliding speed and pressure, led to a decrease in soap friction. At low SRRs and low speeds, friction became more variable which suggests that shear rate influences friction. The fact that soap friction strongly depends on pressure raises the question of soaps exhibiting pressure dependent plasticity. This effect could be the reason for this observed soap film with a constriction near the outlet. The rheology of the soap should also be investigated in this context which could also aid in understanding the effect of slide-roll-ratio, or strain rate, on friction.

The soap formulation plays an important role in the friction behaviour; not only the metal ion of the soap but also the fatty acid content contribute to the soap friction. A

⁴ Implicit industry know-how, personal communication by Stijn de Pauw, Bekaert

borax coating on the disc specimen surface showed slightly improved friction performance, in line with the real wire drawing where borax coating on the wire is used as a lubricant carrier for better lubrication. Tests conducted with different soap particle sizes showed that the grain size did not have a significant effect on friction in contrast to what was found by Byon (55).

These lab tests were complemented with a limited number of tests on a real wire drawing set-up where energy consumption during wire drawing process was measured. The general trends in these tests are in line with those on the ETM. In addition, the residual soap films on the real drawn wires were similar to those on the ETM discs post-test. These observations provide some evidence that the lab ball-on-disc tests with suitable modifications, such as the use of a soap scoop and the use of representative die and wire materials, can be used to study the lubrication performance of powdered wire drawing soaps.

9.2 Nature of Soap Films

The direct observations of the soap films in and around the contact on the high-speed EHD-rig showed that soap films are dynamic with film coverage and thickness changing during the tests even under fixed contact conditions. At times however, especially at high speeds, the soap films resemble a horse-shoe shape that is typical of EHD films with oils. The observed film features allow for some tentative suggestions for soap lubrication mechanisms to be discussed.

The limited attempts at modelling soap films during wire drawing treat soaps as fluid inside the contact and assume hydrodynamic film formation. Present results show that this is a gross oversimplification of the soap film formation mechanisms and is likely to lead to erroneous conclusions. Comparing the formed soap films in the contact during EHD-HS tests to those observed with 'semi-solid' lubricants, there appear to be some similarities between the two, for example with gels, e.g. (100,101), or greases, e.g. (63,102). Similar to what was observed in this work, the nature of the lubricating grease films at low speeds at least, also strongly depends on the type of soap in the greases (103). High speed and high load conditions, and hence high contact temperatures,

appear to aid film formation and lower soap friction.

Thus, an important question was whether the soaps undergo a phase transition caused by high in-contact temperatures due to high sliding and high-pressure conditions. The calculated flash temperatures under the tested conditions are estimated to rise well above 200°C which are certainly within the range where soap melting becomes a possibility. DSC results indicated that the sodium soap used here may indeed show some degree of melting or at least softening. The anhydrous sodium soap transitions into a subneat or neat soap at these temperatures (94,96) which flows easily and resembles a soft turbid gel (104). Likewise, the residual sodium soap film on the ETM disc post-test (e.g. Figure 5-2) was comparable to microscopic images of subneat and neat phases of sodium stearates where the fibres from the curd soap are no longer visible (95). However, it is not clear whether these residual films are obtained due to possible irreversibility of phase transitions of the sodium soap. In general, it seems that for the sodium soap, different transitions can occur throughout the different tests. Consequently, the sliding speed and pressure conditions which affect in-contact temperature rise have a crucial impact on sodium soap films.

However, a constriction near the outlet resembling the horse-shoe shape of a standard EHD fluid film is seen even for 'lower' flash temperature rises at which, for example, the melting temperature of studied sodium-based soap is not reached yet. This may suggest that melting itself is not a necessary condition for effective soap lubrication, a transition to a softened state that exhibits gel-like behaviour may be sufficient.

For the sodium-based soap in particular, a transition into a waxy state might occur at 'lower' temperature rises (below 100°C) as was described by Vold and co-workers (94–96). The observations of in-contact film also showed that some interference fringes appear, implying some translucence of the soaps. Sodium soaps in the waxy phase are considered to be translucent (104).

On the other hand, it seems unlikely for the calcium-based soap (35% FA) to have melted. Therefore, based on the results from DSC measurements and flash temperature calculations for the calcium-based soap (35% FA), it is again dubious if partial or complete melting into a liquid state is even needed for soaps to exhibit this low friction

and to form this lubricating layer. Nonetheless, the high contact flash temperatures are probably still necessary to facilitate those low friction conditions. It could be that the higher in-contact temperatures lead to a softening of the soap particles and therefore, these soap particles are more easily sheared. Calcium soaps are expected to undergo polymorphic transformations at temperatures between 65°C and 195°C (98) which could lead to softer soap particles and thus, result in films with low shear stress. This hypothesis is also plausible for the sodium soap, where a transition into a waxy state can be assumed for temperatures below 200°C, at which sodium soap is considered to be adequately softer than in the curd phase (104).

In addition, observations of particles passing through the contact (chapter 6.2) also suggest that some mechanical effects such as soap particle deformation and compacting, and deposition on the surface also occur – these in themselves may be important mechanisms contributing to soap lubrication.

In general, the nature of soap films is not simple and not universal across different soaps. The low shear stress films could be a combination of softening and/or liquifying of the soaps and mechanical interactions. For deeper understanding of the impact of temperature and the potential effect of phase transitions of soaps on friction, further work is needed. In this context, it could be useful to analyse the crystalline structure of deformed soap particles post-tests.

9.3 *Limitations of Experiments*

In this work, new methods and experimental techniques were used to study friction and lubrication under conditions resembling wire drawing conditions. The tribometers used in this work offer the advantage of measuring friction directly with powdered soaps but also have some limitations. The optical techniques used provide good insights into the nature of the films and potential lubrication mechanisms, yet they come with their own drawbacks.

The ball-on-disc tribometers offer a proven way to perform systematic studies of friction and film forming behaviour of lubricants under controlled conditions. They are therefore

invaluable in establishing the lubrication mechanisms at play for a given lubricant, powdered soaps in the present case. However, with respect to wire drawing, the contact conditions in the ETM and EHD-HS rigs are quite different from the real die-wire contact. Similar trends between energy consumption in wire drawing and friction behaviour on the ETM machine (chapter 7), as well as similarities in residual films on real drawn wires and ETM discs post-tests, give reassurance that ETM tests can capture the primary mechanisms and the obtained trends are representative of those in real dry wire drawing. However, there are a number of differences between the ETM and optical EHD conditions and those in the real wire-die contact. In wire drawing, wire and drawing die form a conformal contact. The objective of this work was therefore more focussed on mimicking the severe conditions in wire drawing which was indeed possible with the tribometer used. The tested pressures lie in the order of magnitude of the range of yield strength of the wire (e.g. (105)). A major drawback of the ETM friction experiments is the limited speed of the ETM rig. Whilst the ETM is able to apply high loads and pressures similar to those in a wire-die contact, it is restricted to speeds of maximum 4 m/s. In industrial practice, much higher speeds up to 25 m/s are reached. In this work, this is mitigated by the use of the high-speed EHD optical rig which is able to impose entrainment and sliding speeds up to 20 m/s.

Limitations specific to the optical technique for in-situ contact observation include the lack of knowledge of refractive indices of the powdered soaps. In their initial state, studied soap powders are opaque but observations on the EHD, as well as on the confocal microscope showed the appearance of interference fringes in the soap film. Knowing the refractive indices of those soap films could be used to give quantitative insights about film behaviour and film thickness in the contact, rather than only qualitative assessment as was done in this research. In addition, the thickness of deposited layers on specimens was estimated to be very thick, from several hundred nanometres to a few microns. The EHD set-up only allows film thickness measurements of a few hundred nanometres. Hence, even if the refractive index of soaps was known, another technique such as fluorescence could be more useful to determine in-contact film thickness for thicker films seemingly produced with powdered soaps.

The UWT machine was used to measure actual drawing stresses when wire was drawn lubricated with dry powdered soaps, but it is not capable of measuring friction directly

during drawing. Moreover, it cannot provide direct insights into the mechanisms at play at the wire-die interface since it does not allow direct visual access to the contact. Nonetheless, the results obtained from the UWT depict the real trends observed in wire drawing and conducted friction tests in this thesis aligned well with the results from the UWT.

The temperature calculations and associated implications were limited to the contact conditions in the lab machines, e.g. in the ETM and EHD contact. The flash temperature rises in the wire-die contact were not studied in the context of this thesis but such a study would help to better compare the nature of lab produced soap films to the soap films on the real wires. In addition, above calculations are only rough estimates of the flash temperature rises in the contact and do not account for the dynamic of the soap films.

In order to fully understand the nature of the films, more information regarding the chemistry of the soaps is needed. Current work was limited to three different soaps where not only metal cation content, but also fatty acid content differ. Hence, no conclusive evaluation concerning the effect of chemical components can be made. In an attempt to mitigate against these issues of using a fully formulated soap, this work also used some simple sodium and calcium stearates, but more work is needed. Moreover, the mechanical properties which again also depend on the soap chemistry have not been studied in this work.

Lastly, the different test set-ups may have significant differences in the entrainment of soap powder into the contact. Likewise, the entrainment of soap powders in the real application might also be different. In the present work an attempt was made to mitigate against any variability in test results introduced by entrainment variability by the use of the soap scoop, but the actual effect of different entrainment arrangements was not studied.

10 Conclusions and Future Work

10.1 Summary of Key Findings

The aim of this research was to study and understand the friction and lubrication behaviour of powdered soaps used in dry wire drawing. In industry, soaps are commonly used as a dry lubricant to produce steel wires up to a thickness of about 1 mm, but the tribological interactions in the wire-die contact lubricated with soaps are insufficiently studied and poorly understood. Since friction measurements in the real application are very difficult, in this work, an ETM ball-on-disc tribometer was used to study the frictional behaviour of typical sodium-based and calcium-based wire drawing soaps under conditions pertinent to the wire-die contact. Deposited films on the ETM specimens and real wires were analysed using optical techniques such as confocal and SEM-EDX microscopy, and Raman spectroscopy. To complement this and aid in understanding the nature of soap films, a high-speed optical EHD machine was used to observe the entrainment of soap powders and soap film formation directly in the contact. Finally, with the help of the industrial sponsors, a real wire drawing machine, referred to as Universal Wire Drawing Test Machine (UWT), was used to measure the energy consumption during dry wire drawing.

The main findings of the present research are:

1. The ETM ball-on-disc tribometer with addition of a soap scoop to avoid starvation in the contact, and careful experimental set-up including filling the chamber with soap, can indeed be used to investigate the frictional performance of typical dry powdered soaps. The results showed that low, stable friction values were achieved with the powdered soaps. Similar trends were observed on the ETM ball-on-disc machine and the real dry wire drawing machine. The wires themselves showed similar deposited layers as the ETM discs post-test. This offers reassurance that the lab-based tests used here are able to capture the primary mechanisms of soap lubrication.
2. Optical EHD technique is also helpful to get first insights into the nature of those films, particularly if it allows for high speeds, such as the high-speed EHD (EHD-

HS) rig used here.

3. Friction measurements on the Extreme-traction machine with typical sodium- and calcium-based wire drawing soaps led to the following findings:

- It was found that soaps generally produce very low friction. COFs are as low as 0.03 for the high fatty acid content (78%) sodium soap and the medium fatty acid content (55%) calcium soap at high sliding speed and high pressure. The low fatty acid content (35%) calcium soap led to slightly higher COFs of 0.04 under the same conditions.
- Soap friction was highly affected by sliding speed and pressure. In general, friction decreased with increasing sliding speed and pressure. High SRRs and high sliding speed resulted in very steady and low friction coefficients whereas at low sliding there was significant variability in measured friction results.
- Based on results of tests with simple sodium and calcium stearates, initial results showed that higher fatty acid content, rather than the type of metal soap, may be responsible for low friction at high speeds and high pressures.
- Borax coatings are used in wire drawing to enhance soap pick-up on the wires and soap lubrication. ETM friction was also measured with borax-coated disc specimens which indeed resulted in slightly lower COFs for a short period of time than with uncoated discs. However, it was challenging to ensure consistency and survival of the borax coating on the ETM discs throughout the tests.
- In the scope of this thesis, different soap particle size distributions ranging from a few microns to a few millimetres were investigated. The results showed that the particle size of soap powders did not seem to have a strong influence on friction.
- Case-carburised specimens were used broadly to avoid large

deformations during the ETM tests. Comparisons with annealed specimens showed no impact of the surface treatment of the specimens on friction.

4. To better understand the lubrication behaviour of soaps, the residual deposited layers on the specimens were analysed post-test using optical techniques such as Raman spectroscopy, confocal and SEM-EDX microscopy. In most cases, relatively thick solid soap films were present on the rubbing track post-test with an estimated thickness ranging from several hundred nanometres to the micron range. The morphology of the soap films varied with the type of soap and type of test, yet the low fatty acid content (35%) calcium soap achieved the thickest films, but also the 'highest' friction compared to the other soaps.
5. A newly developed optical ball-on-disc rig capable of high speeds (high-speed EHD machine) was employed to directly observe soap films in and around the contact. The direct access to the contact allowed to investigate how soap particles were entrained and how they formed a lubricating layer. Results showed that soap films exhibit a very dynamic behaviour with thinner and thicker films, as well as starvation at times. It was found that intermittently, particularly at higher speeds, soaps also produced a film shape resembling the typical horse-shoe shape from an EHD film distribution. This raised the question whether the soaps behaved like a fluid or like a soft gel-like-solid at times under high-speed, high-pressure conditions.
6. In this context, the effect of temperature on soap lubrication was studied. High flash temperature rises, well above 200°C, were predicted in the WC ball on steel disc contact in the ETM at high sliding speeds and pressures. Differential scanning calorimetry measurements showed that soaps undergo some phase transition at these temperatures. The studied sodium soap is assumed to liquify and transform into a neat soap with a viscous behaviour under the test conditions. However, results suggest that calcium soaps are unlikely to melt but become softer and easier to deform under the high sliding and high-pressure conditions. In addition to the temperature effects, the mechanical interactions such as deformation of soap particles, could also contribute to the film formation.

7. In general, this work offers new quantitative friction data for dry wire drawing soaps and new insights into soap lubrication mechanisms. The work can help in developing better models of die-wire contact and in general, in improving the dry wire drawing process.

10.2 Future Work

Although useful in itself, the present work can be extended in multiple ways. Some suggestions for future research in this field include:

- A study with a wider range of soap formulations could reveal new information regarding chemical components affecting friction performance. It would be interesting to identify key components of soap powders that could further improve lubrication. This requires a systematic approach with a series of custom-made soap formulations where only one component is varied at a time. Given that these lubricants are relatively niche and highly specialised products, this is not easy to achieve (an attempt was made in this project), and it would require involvement of a soap manufacturer as well as a dry wire manufacturing company.
- In addition to the isolation of certain chemical components affecting soap lubrication, an attempt should be made to determine what factor is primarily responsible for observed dynamic behaviour of the soap film. This could include further studies of variable entrainment due to different size distributions of soap particles or contact conditions. Moreover, mechanical properties of particles, e.g. hardness and stiffness, could have a major effect on soap deposition and film formation.
- It would also be helpful to provide more fundamental and extensive studies of the tribological interactions in the actual wire-die contact using set-ups such as the UWT, which was used only for a limited number of tests in this study. In particular, the transfer of optical techniques to the wire drawing set-up would be helpful to directly observe and perhaps quantitatively measure the soap film in

the wire-die contact. These studies should also include the assessment of different roughnesses of specimens and their effect on lubrication and friction performance.

- The temperatures in the contact seem to be a key factor in tribological performance of soaps. Conducted experiments showed that at conditions that lead to high temperature rises, e.g. high sliding speeds and high pressures, friction was very low. First results showed some temperature dependent change in the state of the soap, but it is not clear if the soap is transitioning into some highly viscous crystal liquid followed by melting, at least for the sodium soap, or any other state. Perhaps, the soaps remain completely solid, but the particles are softened and thereby easier to form a soap layer on the surfaces. The soaps might also just form a mechanical adhesive film.

Based on this, it is crucial to establish if some soaps undergo irreversible changes at elevated temperatures. Analysis of the crystallographic state of the soaps after tests could give some indications about temperatures reached in the contact.

- In view of the effect of temperature on soaps, earlier presented results are only rough estimates of in-contact temperatures. For an improved interpretation of the results, it could be interesting to measure the in-contact temperature using for example an infrared camera, e.g. (106). If the technique works with powdered soaps in a ball-on-disc machine, this set-up might also be adapted to the real wire drawing machine.
- Another aspect to consider is high speed friction tests. Earlier presented experiments showed a decrease in friction with increasing sliding speeds up to 4 m/s which did not seem to reach a plateau yet. Literature on wire drawing experiments also indicated a further decrease in drawing stress up to speeds of 15 m/s (9). Present work did use a high-speed optical EHD rig to observe soap films at high speeds greater than 10 m/s but it would obviously be interesting and of practical importance to also study friction at high speeds representative of wire drawing speeds.

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- Finally, in terms of the optical machine used, the set-up could be further adjusted to use fluorescence given the apparently thick soap films. Fluorescence techniques are useful to study thicker films, e.g. (107), and could be used in a ball-on-disc set-up similar to the experimental work by Jeffreys *et al.* (108).

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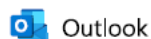
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MTM Grease Scoop Image

Von Kieran Nar <kieran.nar@pcs-instruments.com>

Datum Di, 29.10.2024 17:07

An Schlichting, Marie-Louise M <m.schlichting20@imperial.ac.uk>

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Hi Marie-Louise,

Thank you for reaching out to PCS Instruments.

We are happy for you to use the MTM grease scoop image from your original email in your PhD thesis.

Hope the writing is going ok - thanks,
Kieran

Kieran Nar

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