Transport Phenomena in Multi-phase Media: Slurries, Foams,

Boiling Bubbly Media and Pulps

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THESIS

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SUMMARY

The aim of this work is to study experimentally and theoretically the transport phenomena of multi-phase materials: slurries, foams, boiling bubbly media and pulps.

Foam is used in many industrial applications. Foam is added to gypsum slurries to make lighter wallboards. To produce high quality low-cost wallboards, understanding of rheology of gypsum slurries with foam is important. Foam content in slurries could significantly affect the rheological behavior and the results of this work quantified these effects. In particular, it was shown that the power-law, Ostwald-de Waele rheological constitutive equation is applicable in such cases and its rheological parameters (the exponent and consistency index) were measured as the function of foam content.

A theoretical model of gravitational foam drainage as a consolidating continuum was proposed. The general model was applied to foam drainage in a gravity settler. It was predicted that liquid drainage from foam in a gravity settler begins with a slow drainage stage. Next, a second stage with a faster drainage process sets in where the drainage rate doubles compared to the initial stage. The experiments conducted in the PhD research of D. Pelot corroborated these predictions.

The approach based on the ideas of the consolidation theory was applied to predict the evolution, stability and approach to a steady-state configuration of threads of Pantene foams. The experiments with threads of Pantene foams at different concentrations were conducted and compared to the theoretical predictions. The comparison showed that relatively short 8% Pantene foam threads approach a steady state configuration, as predicted by the theory. The maximum difference of the steady-state thread profiles predicted theoretically from those measured experimentally was about 3-11%.

Pool boiling on nano-textured surfaces was studied experimentally and theoretically for ethanol and water as working fluids. The nano-textured surfaces represented copper platelets covered with copper-plated electrospun nanofibers. In addition, for comparison, pool boiling on the corresponding bare copper surfaces was experimentally studied. The results revealed that the heat flux and heat transfer coefficient in pool boiling on the nano-textured surfaces were about 3-8 times higher than those on the bare copper surfaces. This result stems from the fact that nano-textured surfaces promote bubble growth by increasing the average fluid temperature surrounding growing bubbles, as the experimental and theoretical results of this thesis show. Hence, nano-textured surfaces comprised of copper-plated nanofibers facilitate bubble growth and, thus, increase bubble detachment frequency. On the other hand, the critical heat flux (CHF) on the nanotextured surfaces was found to be very close to its counterpart on the bare copper surfaces. On the other hand, the heat flux on the nano-textured surfaces in transition boiling was significantly higher than that on the bare copper surfaces, since the presence of nanofibers prevented bubble merging and delayed formation of vapor film.

Cracking of USG ceiling tile materials (water saturated pulps) was studied experimentally and theoretically. To observe the cracking phenomena, three point bending tests were performed on vacuum drained materials with 83% water. From the bending test results, stress-strain relations were established along with corresponding mechanical properties such as Young's modulus, the yield stress, and cracking stress and strain. The tests showed Young's modulus 705 kPa, yield stress 2.4 kPa, stress at

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cracking 12 kPa, and cracking strain 10%. Furthermore, a plausible physical mechanism was proposed and the corresponding theory was developed. The theory showed that cracking occurs at 35 mmHg vacuum pressure, which is in a fair agreement with the actual cracking position at the industrial conveyor belt.

1. INTRODUCTION

Complex slurries, foams and nano-textured materials are attractive for potential applications in several areas, e.g. in mechanical and chemical engineering and materials science, and such industries as construction, cosmetics and food, as well as textiles and energy. The analysis of the experimental data and establishment of a suitable physical model and constitutive equations describing transport phenomena in these materials are important steps for their practical application.

In this thesis, several inter-related types of transport phenomena were investigated experimentally and theoretically. Momentum transport of gypsum slurries with foam (Chapter 4) was studied by measuring shear viscosities with different foam contents. The shear viscosities were processed to find the appropriate rheological parameters in the framework of the power-law (Ostwald-de Waele) model. Foam itself was also a research subject in the present thesis. In particular, foam drainage and consolidation were studied in Chapter 5, and foam behavior in the elongational and shear flows were tackled in Chapters 6 and 7. Foaming results in conglomerates of bubbles. Another situation where significant bubbling takes place is pool boiling. In Chapter 8 bubbling and fully developed pool boiling on nano-textured surfaces was investigated experimentally and theoretically. A solid-like equivalent of foams is found in paper pulp used to form ceiling tiles. In this case the liquid of foams is replaced by soft solid particles forming a skeleton, and the air and vapor of foams are replaced by liquid. These materials are studied experimentally and theoretically in Chapter 9 with a particular interest in the overall consolidation and cracking.

2. BACKGROUND AND LITERATURE SURVEY

2.1. Transport phenomena in gypsum slurries with and without foam

Transport phenomena are associated with mass, momentum, species and energy transfer in continua. The materials of interest can be very complicated, non-uniform and subjected to different internal and external forces. The overall governing equations describing transport phenomena include the mass and momentum balance equations, as well as the species transport and the energy balance equations. However, these equations are not closed unless the constitutive equations describing the internal stresses and fluxes are formulated, which is a non-trivial task in the case of complex fluids, for example non-Newtonian liquids, suspensions, emulsions and slurries. A part of mechanics called rheology deals with formulating the constitutive rheological equations for complex fluids (Frederickson, 1964; Coleman et al., 1966; Ferry, 1966; Astarita and Marruci, 1974; Tanner, 2000). The complex material of interest in the present work is gypsum slurries used to make wallboards.

Gypsum is a soft mineral material composed of calcium sulfate dehydrate (CaSO4•2H2O). In the process of wallboard production, several additives to gypsum stucco are used such as water, an accelerator, dispersant, and soap foam. Gypsum slurries solidify at extreme pace due to rapid evaporation of water. The study of shear and elongational rheology of gypsum slurries without foam was conducted by Sinha-Ray et al. (2011). They measured shear viscosities of gypsum slurries using a shear viscometer and revealed pseudoplastic (shear thinning) rheological behavior. Moreover, they showed that the pseudoplastic behavior is consistent with the elongational behavior of gypsum slurries,

thus establishing the tensorial structure of the rheological constitutive equation. Soap foam is a very important additive to form bubbles in gypsum slurries and ultimately to make voids in the solidified wallboards to make them lighter. The added foam at different concentrations changes rheological behavior of gypsum slurries. Therefore, there is a need to understand the rheological behavior of gypsum slurries incorporating foam in order to maximize the efficiency of the processing operations.

2.2. Foam characterization and stability

Foam is important not only for the construction industry but also for many other applications such as food and beverage industries, personal care industry, textile industry, fire retardants, general polymers/plastics, oil recovery and mineral flotation processing (Prud'homme and Khan, 1996). Foam mechanics have attracted researchers for a long time beginning with the seminal works of Plateau in the mid-19th century. The factors controlling foam changing, patterning and stability are drainage, coarsening (the interbubble gas diffusion caused by the pressure difference across the separating lamellae), and lamella thinning and rupture (Kornev et al., 1999). Regarding drainage, some authors claim that it is caused by gravity (Kornev et al., 1999), whereas others believe that the gravity effect is negligible and drainage is caused by capillary suction to the Plateau borders (Ranai et al., 1993). In the experiments with foam drainage, it was found by different authors that only certain factors influence the drainage rate and the volume of liquid drained, although there are disagreements. For example, Saint-Jalmes and Langevin (2002) found that different gases used in creating foams can change the drainage rate and the overall drainage time. In Germick et al. (1994) it was concluded that foam drainage was faster and the volume of liquid drained was larger for smaller bubbles and larger initial heights, whereas in Ramani et al. (1994) it was stated that the drainage rate was slower for larger initial heights and the maximal rate was the same regardless of height. However, factors such as column diameter and column material have no effect on drainage rate or drainage time according to Ramani et al. (1994). In addition, Magrabi et al. (2001) found that the time to full drainage was inversely related to the liquid fraction.

Several empirical equations were proposed to fit foam drainage data (Ross, 1943; Ramani et al., 1994; Kornev et al., 1999; Keohler et al., 2000). One of the first empirical foam drainage equations was proposed in Ross (1943). This equation describes the fraction of liquid in the foam by an exponential function involving the current and characteristic time, with the latter being a fitting parameter. Its popularity is probably the result of its simplicity. An equation that predicts the liquid drained as a function of time was also proposed in Ramani et al. (1994). However, in addition to the liquid volume fraction, this equation involves such parameters as bubble diameter, solution viscosity, radius of Plateau border (Kornev et al., 1999) and the lamellae thickness in the foam, which cannot be easily established in ordinary foams.

Foam longevity can be enhanced by adding stabilizers of 6 different types (Prud'homme and Khan, 1996):

(i) Organic compounds (fatty alcohol, fatty acids, alkanol amides, amine oxides, and various other surfactants). These organic stabilizers typically stabilize foam by enabling the surfactant molecules to form a closely packed film that exhibits more elasticity and more resistance to drainage, and consequently is less susceptible to external disturbances (Miles et al.,

1944; Miles and Ross J, 1944; Rosen et al., 1964; Zhang et al., 2005; Famuea et al., 2011).

(ii) Electrolytes (salts). An electrolyte can stabilize the foam generated by anionic surfactants by reducing critical concentration for micelle formation thus decreasing the repulsion between the head groups at the air-liquid interface (Lessardl and Zieminski, 1971; Christenson and Yaminsky, 1995; Shao and Van de Ven, 1988; Pashley and Craig, 1997; Karakashev, 2001;).

(iii) Polymers (polyvinyl alcohol, sodium alginate, sodium dodecyl sulfate-poly (ethylene oxide)). Water-soluble polymers can stabilize foams by increasing either surface or bulk viscosity of the film, thereby increasing the film elasticity or decreasing the film drainage rate (Goddard and Hannan, 1976; Kwak, 1998; Alargova et al., 2004; Duan et al., 2004).

(iv) Biopolymers (proteins, agar-agar, gelatin, albumin, complexes formed between phospholipids and proteins). The stabilization of the air-liquid interface by biopolymers has been ascribed to the adsorption of the protein at the interface, leading to the formation of a cohesive and viscoelastic film, a condition required for foam stability (Lee et al, 1997; Shyamkuwar et al., 2010; Murray et al., 2011).

(v) Liquid crystals (ternary system, containing water/i-amine octane/p-xylene). Liquid crystalline phases can stabilize foams by enhancing viscosity of film liquid, which retards the drainage rate and increases surface viscosity of the interfacial film due to the adsorption of liquid crystals at the air-liquid interface (Aramaki et al., 2001; Shrestha et al., 2006; Yan et al., 2011).

(vi) Micro and nanoparticles, which tend to self-assemble at the bubble surfaces and shield them, so they prevent bubble coalescence in foam or increase the apparent viscosity of the liquid. Micro- and nanoparticles form mineralized foams (e.g. silica, alumina, latex, talc, quartz, soot). The latter is similar to the so-called Pickering emulsions. It is known that smaller (e.g. submicron) hydrophilic particles are good foam stabilizers (Du et al., 2003; Dickenson et al., 2004; Kostakis et al., 2006; Fujii et al., 2006; Gonzenbach et al., 2006, 2007; Sherstha et al., 2007; Wege et al., 2007; Vignes-Adler and Weaire, 2008; Hunter et al., 2008; Karakashev et al., 2011; Wong et al., 2011).

The ultimate goal of all six approaches mentioned above is to stabilize bubbles growing or pre-existing in a foaming matter. To prepare stabilized foams, organic compounds which are good foaming agents are usually used as surfactants, and the other additives are mixed with them.

2.3. Pool boiling

Pool boiling is associated with the development of vapor-filled foam-like matter near the heated surface. It is known as a very effective mechanism of two-phase heat transfer due to its high heat flux values. The regimes of pool boiling can be divided on the boiling curve as free (or natural) convection boiling, nucleate boiling, transition boiling, and film boiling (Nukiyama, 1966). The boiling curve is shown in Fig. 2.1. When the bulk liquid starts boiling, the heat is transferred from solid surface to the bulk liquid by natural convection. If the superheat temperature (ΔT_{sat}) is beyond point A, bubbles are formed at some places on the solid surface, which are called nucleation sites. This regime is denoted nucleate boiling. It combines two different parts. The first one is where isolated bubbles which do not interact with each other are formed. As the superheat temperature moves beyond point B, the bubble formation rate increases and they interact with each other, which is denoted as the slugs and columns regime. As the superheat temperature further increases, the heat flux reaches the critical heat flux (point C in Fig. 2.1) which corresponds to the maximum heat flux. Beyond the critical heat flux, bubbles merge and form a vapor film over the solid surface. This hinders heat transfer from the solid surface to the bulk liquid and it is denoted as transition boiling. As the superheat temperature continues to increase, it reaches the minimum heat flux (point D in Fig. 2.1) known as the Leidenfrost temperature. Beyond that temperature, a stable vapor film separates the solid surface from the bulk liquid, and this situation is denoted as the film boiling regime. In this regime, the radiation heat transfer is dominant across the vapor film, and the heat flux increases once again as the superheat temperature increases.



Fig. 2.1. Standard pool boiling curve for saturated water (Thermal Fluid Centrals, 2010).

Among these scenarios, nucleate boiling is considered to be the most desirable heat transfer regime, since it is associated with high heat fluxes at low superheat surface temperature, which is attractive for such practical applications as cooling of electronics, boilers, nuclear and chemical reactors, and refrigeration systems. However, there exists a critical value of the heat flux called critical heat flux (CHF) at which nucleate boiling transforms into film boiling. The latter involves heat transfer through coolant vapor rather than through the liquid coolant itself, which is a heat transfer mechanism. There were multiple efforts to increase CHF and boiling heat transfer coefficient (BHT), which is by definition the heat flux divided by the corresponding superheat temperature. In particular, boiling on roughened surfaces was studied in the early works on pool boiling enhancement (Jakob, 1949; Courty and Foust, 1955; Kurihari and Myers, 1960; Berenson 1962). It was found that roughened surfaces increase the BHT coefficient, which stems from an increased area density of nucleation sites. However, the CHF was about the same as the one for smooth surfaces (Berenson, 1962). Microporous coatings to enhance pool boiling were studied in (Bergles and Chyu, 1982; Chang and You, 1997; Rainey and You, 2000; Liter and Kaviany, 2001; Kim et al., 2002; Li and Peterson, 2010). In particular, Li and Peterson (2010) sintered microporous surfaces and found that CHF on them strongly depends on the coating thickness, volumetric porosity, and pore size. They attributed the improvement of heat transfer in their experiments to an increased effective heat transfer area. It was also found in Li and Peterson (2010) that when the coating thickness reaches approximately 2 mm, CHF reaches a maximum, which was twice that of the corresponding bare surface. Volumetric porosity does not greatly affect CHF value. On the other hand, CHF increases with increasing pore size up to 250 µm (Li and Peterson,

2010). Recently, the effect of nanoparticles suspended in liquid on pool boiling was studied (You et al., 2003; Das et al, 2003; Vassallo et al., 2004; Wen and Ding, 2005; Kim et al., 2007; Kim and Kim, 2009, Kwark et al., 2010). The authors of You et al. (2003) investigated the effect of alumina (Al_2O_3) nanoparticles from 0 g/l to 0.05 g/l dispersed in water and reported that CHF can be doubled at larger than 0.005 g/l, but the value of CHF did not change much above 0.01 g/l. However, the heat transfer coefficient was about the same as without nanoparticles. Kim et al. (2007) studied several nanoparticles such as alumina (Al₂O₃), zirconia (ZrO₂), and silica (SiO₂) in water and the results showed that the CHF was increased by 52% with alumina, by 75% with zirconia, and by 80% with silica. An enhancement of CHF in nucleate boiling in the presence of nanoparticles is commonly attributed to an increase in surface wettability (Kim et al., 2007; Kim and Kim, 2009; Kwark et al., 2010). However, the reports on the effect of nanoparticles on BHT show discrepancies. Some studies reveal BHT deterioration (Bang and Chang, 2005; Kim et al., 2007). In Kim et al. (2007) the BHT coefficient decreased due to: (i) a build-up of the nanoparticle layer, which reduces heat transfer from the metal surfaces in pool boiling, and (ii) a reduction of nucleation site density caused by a decrease in the contact angle. Some other studies show no difference (You et al., 2003). On the other hand, Wen and Ding (2005) claim that nanoparticles can enhance BHT up to 40% due to nanoparticle migration within homogeneous nanofluids. No comprehensive theory explaining the changes in BHT and CHF due to the presence of nanoparticles in pool boiling was proposed, however in Kim et al. (2007) the experimental data were discussed in the framework of such theories as the macrolayer dryout theory, hot/dry spot theory, and bubble interaction theory. The uses of carbon nanotubes (CNTs) have been

studied in relation to pool boiling during the last years because of their good conductivity and large area to volume ratio (Ahn et al., 2006; Ujereh et al., 2007; Park and Jung, 2007; Park et al., 2009; Mchale et al., 2011). CNT-coated surfaces increase BHT and CHF compared to smooth surfaces. In particular, Ujereh et al. (2007) studied pool boiling of FC-72 and reported 450% and 40% enhancement of maximum BHT and CHF, respectively, on fully CNT-coated silicon surfaces compared to a smooth silicon surface. They also reported a 30% and 6% enhancement of maximum BHT and CHF, respectively, on fully CNT-coated silicon surfaces compared to smooth copper surface. The enhancement due to the CNT coatings is because of the initial roughness difference between silicon and copper. Actually, the values of BHT and CHF of CNT-coated surfaces of silicon and copper turned out to be almost the same. Park et al. (2009) studied pool boiling of water. They dispersed CNTs in water at different CNT volume concentration and showed that BHT decreases as the CNT concentration increases, but the CHF was increased by 200% at 0.001% CNT concentration. Still, the reasons for the CNT-related enhancement in pool boiling are not clear. In particular, it is not clear whether changed wettability or surface area enhancement or surface roughness play the main role.

2.4. Cracking in soft materials

Cracks are observed in soft materials such as clays, soils, and slurries when they are desiccated and shrunk. When materials are used for practical purposes, cracking is an important issue since it makes materials weaken. Many researches have tried to elucidate cracking mechanisms of soft materials (Morris et al., 1991; Komatsu and Sasa, 1997;

Colina and Roux, 2000; Yesiller et al, 2000; Thusyanthan et al., 2007; Geohring, 2009; Atique and Sanches, 2011; Kodikara and Costa, 2013). It was found by different authors that certain factors influence cracking, for example, for the extent and development of desiccation cracking, there are many common factors which can be named. For example, Colinan and Roux (2000) found that clay content and mineralogy, soil thickness, surface configuration, rate of drying, total drying time are all important for cracking. Morris et al. (1992) found that negative pore water pressures (suction) which develops in a soil during drying, the elastic properties of the drying soil, moisture content, confining pressure, temperature, and cycles of wetting and drying all affect desiccation cracking. Generally drying causes inhomogeneous shrinkage and a crack is initiated when stresses exceed the soil strength. The soil strength is dependent on the moisture content of the soil. To measure solids strength, tensile tests are widely used. However, when materials are so soft or weak that they can not be held in a clamp, the material strength can be found by a bending test (Thusyanthan et al., 2007).

3. RESEARCH DESIGN AND OBJECTIVES

To analyze transport phenomena in bubbly media (gypsum slurries, pure foams, and boiling liquid over nano-textured surfaces), several different experimental and theoretical tasks have to be performed. These include:

(i) Rheological characterization of gypsum slurries with different foam contents.

(ii) Theoretical study of foam consolidation and drainage.

(iii) Experimental and theoretical study of foam stability.

(iv) Study elongation and shear rheology of foam.

(v) Experimental and theoretical study of pool boiling over nano-textured surfaces.

(vi) Experimental and theoretical study of cracking of porous multiphase materials.

3.1. Study of rheology of gypsum slurries with foam

Gypsum is massively used in the construction industry to form wallboards. In the process of making wallboards, several additives to gypsum stucco are used such as water, an accelerator, dispersant, and soap foam. Among them soap is a very important additive because it introduces bubbles in the slurries which ultimately produce voids and make products lighter.

The objective of the present work is to elucidate rheological behavior of gypsum slurries with foams and measure the corresponding rheological parameters using shear experiments at different shear rates. The resulting rheological constitutive equation can be used in the framework of computational fluid dynamics to optimize mixing of gypsum slurries with foam in real gypsum wallboard production lines.

3.2. Theoretical investigation of foam consolidation and drainage

This topic is devoted to study of foam consolidation and drainage. The factors affecting foam consolidation and drainage and the physical mechanism of foam drainage are investigated. The objective of this work is to establish a proper physical model describing foam drainage in gravitational settlers. Foam is considered in the framework of the consolidation theory and the predictions are compared to the experiments on foam drainage conducted by another PhD student, David Pelot.

3.3. Theoretical and experimental investigation of foam threads

The objective of this work is to observe and explain the intrinsic behavior of foam threads. It is demonstrated that threads of catenoidal shape are formed under certain conditions. The theoretical predictions are compared to the experiments with foam threads in the elongational rheometer. The effect of a water-soluble polymer, Polyethylene oxide (PEO), on foam thread stability is also investigated.

3.4. Study of elongational and shear rheology of stable and unstable foams

The focus of this part is on stable and unstable foams and their behavior in the two limiting benchmark flows: shear and elongation. To make the foam, a liquid soap is mixed with water. There are two different types of soaps used in practice: unstable and stable foams. The unstable foam creates larger bubbles, since the foam is unstable and bubble coalescence is relatively easy. Bubbles in the stable foam do not easily coalesce.
The elongation and shear rheological tests are done using unstable and stable foams with different air content and in combinations of stable and unstable foam with the same air content. The experimental data are interpreted using the power-law rheological constitutive equation.

3.5. Theoretical and experimental study of the effect of nano-textured surface on pool boiling

The question of interest in this part is to what extent metal-plated nano-textured surface will affect heat flux and pool boiling over them. The aim of the present work is in the experimental evaluation of the effect of nano-textured electrospun coatings on the enhancement of the Boiling Heat Transfer (BHT) coefficient and Critical Heat Flux (CHF) in pool boiling of water and ethanol. In addition, the development of a novel theory explaining the role of nano-textured surfaces in pool boiling is tackled.

3.6. Theoretical and experimental study of cracking mechanism under vacuum drying of multiphase humid materials

In the process of manufacturing of ceiling tiles, ceiling tile materials are used. These materials are humid paper pulp slurries with the geometrical structure resembling that of gypsum slurries and bubbly media. Drying of ceiling tiles involves vacuumization, which has its own drawback, namely cracking. Cracks weaken ceiling tiles and are undesirable. The objective of this work is to find the cause of cracking, understand its mechanism and develop a proper physical model to explain this phenomenon and search the ways to eliminate it.

4. SHEAR RHEOLOGY OF GYPSUM SLURRIES AT DIFFERENT FOAM CONTENTS

4.1. Introduction

In the present work, shear rheology of gypsum slurry at different foam contents is studied since soap foam is added to gypsum stucco before mixing and it is important to evaluate how the presence of foam affects processing. The experimental part is described in section 4.2 and the corresponding theoretical background is given in section 4.3. The results are presented and discussed in section 4.4, and conclusions are drawn in section 4.5.

4.2. Experimental

All the materials used in this experiment were supplied by United States Gypsum Corporation (USG). The stucco used in this work was Southard at 75 water stucco ratio (75 WSR), 68 WSR, 75 WSR fixed irrespective of foam content (75 WSR-FI), and 70 WSR-FI. Also, 0.125 wt% of CSA (climate stable accelerator) and 0.25 wt% of dispersant was blended with the stucco. In addition, 0.5 wt% of 50:50 stable: unstable blend was used for soap foam, and the air flow rate was maintained constant at 1.5 liter per minute at the soap flow rate of 120 g/min. A stucco batch size of 400 g was used up to air contents of 60% v/v. The foam addition time that was required to achieve higher air contents was greater than the mixing procedure had allowed for. Therefore, the batch size was then decreased to 80 g in order to accommodate the higher air contents. The gypsum and foam mixing protocol is presented in TABLE 4.1. After the mixing protocol was completed, a syringe was used to draw the slurry from the mixing bowl and put it into the shear viscometer (TA instruments AR 2000ex) cylinder. A continuous ramp procedure was performed from the shear rate of 0.5 s^{-1} to 800 s^{-1} in 20 seconds. Another slurry sample was used for weighing, to measure slurry density.

TABLE 4.1

GYPSUM SLURRY AND FOAM MIXING PROCEDURE

Time (seconds)	Procedure
0	Stucco is added to the gauging water.
15	Mixer is turned on.
15 - 40	Foam is added as necessary during this time.
40 - 45	Foamed slurry is mixed for an additional 5 sec for better blending.
45	Mixer is turned off and slurry is put in the shear viscometer.

4.3. Theoretical Background

The Gypsum slurries are non-Newtonian fluids as shown in Fig. 4.2, so the first rheological constitutive equation to be applied to them should be the Ostwald-de Waele power law (Astarita and Marrucci, 1974)

$$\boldsymbol{\sigma} = -p\mathbf{I} + \boldsymbol{\tau}, \quad \boldsymbol{\tau} = 2\mathbf{K} \left[2\mathrm{tr} \left(\mathbf{D}^2 \right) \right]^{(n-1)/2} \mathbf{D}$$
(4.1)

where σ is the stress tensor, τ is the stress deviator tensor, p is pressure, D is the rate-ofstrain tensor, I is unit tensor, K is the consistency index and n is the exponent (the flow behavior index).

In simple shear flows Eq. (4.1) reduces to

$$\mu_{\text{shear}} = \mathbf{K} \dot{\gamma}^{n-1} \tag{4.2}$$

where μ_{shear} is equal to $\mu_{\text{shear}} = \tau_{xy} / \dot{\gamma}$, with τ_{xy} being the shear stress, and $\dot{\gamma}$ the shear rate. Simple shear flows in rheometry are typically employed at constant shear rate (which values can be chosen different). Then, measurements of shear viscosity are conducted after transients faded. The shear rheometer in the present experiments was operated, however, with a gradually increasing shear rate. Therefore, the value of $\dot{\gamma}$ was not set in the present experiments but was linearly increasing in time. The interpretation of such experimental data requires a fully transient description which is provided below. The momentum balance equation for shear slurry flow under the assumption that the power law model (4.1) is applicable is given by

$$\rho \frac{\partial u}{\partial t} = \frac{\partial}{\partial y} \left[K \left(\frac{\partial u}{\partial y} \right)^n \right]$$
(4.3)

where u(y) is the velocity profile, and y is the coordinate normal to the wall, ρ is the density and t is time. Note that the shear stress corresponding to Eq. (4.1) in this case is given by

$$\tau_{xy} = K \left(\frac{\partial u}{\partial y}\right)^n \tag{4.4}$$

and the effective shear viscosity corresponding to Eq. (4.2) by

$$\mu_{\text{shear}} = K \left(\frac{\partial u}{\partial y}\right)^{n-1}$$
(4.5)

The initial and boundary conditions imposed are as following

$$t = 0 u = 0 (4.6) y = h, u = At (4.6) (4.$$

where h is the gap and A is the acceleration.

The problem (4.3) and (4.6) was solved numerically by the method of finite differences following Sinha-Ray et al. (2011). Consider the simple shear transient experiments in full detail.



Fig. 4.1. Sketch of the velocity profile changes in the transient simple shear flow.

Equations (4.3) and (4.6) can be rewritten in the following form where the asterisks denote dimensional parameters

$$\rho \frac{\partial u^*}{\partial t^*} = \frac{\partial}{\partial y^*} \left[K \left(\frac{\partial u^*}{\partial y^*} \right)^n \right] = K \left(\frac{\partial u^*}{\partial y^*} \right)^{n-1} \frac{\partial^2 u^*}{\partial y^{*2}}$$
(4.7)

$$t^{*} = 0 u^{*} = 0$$

$$t^{*} > 0 \begin{cases} y^{*} = 0, u^{*} = 0 (4.8) \\ y^{*} = h, u^{*} = At^{*} \end{cases}$$

Equations (4.7) and (4.8) can be transformed into dimensionless form using the following scales

$$t^* \rightarrow T$$

$$u^* \rightarrow AT$$

$$y^* \rightarrow h$$
(4.9)

where T is the duration of the experiment and A is the acceleration.

Then the Eq. (4.7) becomes

$$\frac{\partial u}{\partial t} = M \left(\frac{\partial u}{\partial y}\right)^{n-1} \frac{\partial^2 u}{\partial y^2}$$
(4.10)

where M is a dimensionless parameter $(M = KnA^{n-1}T^n / (\rho h^{n+1}))$.

The initial condition and boundary conditions (4.8) become in the dimensionless form

$$t = 0 u = 0 (4.11) y = h, u = t (4.11) (4.11$$

For the numerical solution, Eq. (4.10) is discretized using the finite difference implicit method as follows

$$\frac{\mathbf{u}_{i}^{m+1} - \mathbf{u}_{i}^{m}}{\Delta t} = \mathbf{M} \left(\frac{\mathbf{u}_{i+1}^{m} - \mathbf{u}_{i-1}^{m}}{2\Delta y} \right)^{n-1} \frac{\mathbf{u}_{i+1}^{m+1} + \mathbf{u}_{i-1}^{m+1} - 2\mathbf{u}_{i}^{m+1}}{\Delta y^{2}}$$
(4.12)

where i is a node location and m is a time.

Rearranging Eq. (4.12), we obtain

$$au_{i-1}^{m+1} + bu_{i}^{m+1} + cu_{i+1}^{m+1} = f$$
 (4.13)

where
$$a = \Delta t M((u_{i+1}^{m} - u_{i-1}^{m})/(2\Delta y))^{n-1}/(\Delta y)^{2}$$
,
 $b = -1 - 2\Delta t M((u_{i+1}^{m} - u_{i-1}^{m})/(2\Delta y))^{n-1}/(\Delta y)^{2}$, and
 $c = \Delta t M((u_{i+1}^{m} - u_{i-1}^{m})/(2\Delta y))^{n-1}/(\Delta y)^{2}$.

The system of Eqs. (4.13) can be solved by tri-diagonal matrix algorithm (TDMA) and this algorithm was coded using Fortran. The results were compared to the experimental data to determine the corresponding values of K and n.

4.4. Results and Discussion

4.4.1. Shear flow of 68 WSR and 75 WSR slurries

The gypsum slurries used had different foam contents which ranged from 0% to 100% (volume) with a 10% v/v increment. Several trials were done for each slurry composition to acquire reliable statistics. The results demonstrated a similar trend with 68 WSR compared to 75 WSR, namely, that an increase in foam content causes a decrease in shear viscosity at any shear rate, except the transition from 90% v/v to 100% v/v foam, as shown in Fig. 4.2. The slopes of all the flow curves in Fig. 4.2 are similar but not exactly identical. For pure foam, the slope is quite different from the other cases.





Fig. 4.2. The shear viscosity vs. shear rate for all compositions at 75 WSR (a) and 68 WSR (b). The results correspond to an average of four trials.



Fig. 4.3. The match of the shear viscosity between the theoretical results and experimental results for 40% v/v foam of 75 WSR. The value of n is 0.25 and K = 200 (g/cm ×s²⁻ⁿ).

The results on the density values and the rheological parameter values of 75 WSR and 68 WSR slurries are collected in TABLE 4.2. They show that the values of the exponent n vary from 0.05 to 0.47. The values of K vary from 901 to 23, with the decrease corresponding to an increase in foam content. The rheological parameters for the whole range of foam content were interpolated using polynomial curve fitting (for both K and n). For both n and K values the 6th or 7th order polynomial were used as described in Figs. 4.4 and 4.5. It is shown that the polynomial interpolations are in good agreement with the processed n and K values. For the exponent n, the overall trend shows that the higher the foam content, the larger the n value though there are some selfconsistent fluctuations. For the consistency index K, the overall trend shows that the higher the foam content, the smaller are the values of K. The values of the coefficients of the polynomials are presented in the polynomial functions in TABLE 4.3. To have a good match between the data points and the polynomial functions, 4 digits of the coefficient were required for n and 5 digits of the coefficients were required for K.

TABLE 4.2

RHEOLOGICAL PARAMETERS OF GYPSUM SLURRIES FOR 75 WSR AND 68

Volume % Foam	75 WSR			68 WSR		
	Density (g/cm ³)	n	$\frac{K}{(g/cm \times s^{2-n})}$	Density (g/cm ³)	n	$\frac{K}{(g/cm\times s^{2-n})}$
0	1.573	0.05	901	1.61	0.05	865
10	1.429	0.20	483	1.45	0.10	428
20	1.252	0.13	482	1.30	0.17	242
30	1.109	0.03	495	1.15	0.03	402
40	0.963	0.25	200	0.99	0.05	222
50	0.794	0.33	99	0.84	0.07	205
60	0.653	0.24	127	0.69	0.25	63
70	0.434	0.18	59	0.54	0.17	81
80	0.316	0.27	70	0.38	0.25	48
90	0.180	0.33	40	0.23	0.32	38
100	0.076	0.41	40	0.08	0.47	23

WSR WITH DIFFERENT FOAM CONTENTS



Fig. 4.4. The polynomial curve fitting for exponent n (a) and consistency index K (b) for 75 WSR slurry.



Fig. 4.5. The polynomial curve fitting for exponent n (a) and consistency index K (b) for 68 WSR slurry.

The polynomial interpolation functions with foam volume content c as a variable are listed below

$$\mathbf{n}(\mathbf{c}) = \mathbf{a}_0 \mathbf{c}^6 + \mathbf{a}_1 \mathbf{c}^5 + \mathbf{a}_2 \mathbf{c}^4 + \mathbf{a}_3 \mathbf{c}^3 + \mathbf{a}_4 \mathbf{c}^2 + \mathbf{a}_5 \mathbf{c} + \mathbf{a}_6$$
(4.7)

$$\mathbf{K}(\mathbf{c}) = \mathbf{b}_0 \mathbf{c}^7 + \mathbf{b}_1 \mathbf{c}^6 + \mathbf{b}_2 \mathbf{c}^5 + \mathbf{b}_3 \mathbf{c}^4 + \mathbf{b}_4 \mathbf{c}^3 + \mathbf{b}_5 \mathbf{c}^2 + \mathbf{b}_6 \mathbf{c} + \mathbf{b}_7$$
(4.8)

where the coefficients are listed in TABLE 4.3.

TABLE 4.3

THE INTERPOLATION COEFFICIENTS CORRESPOND TO EQS. (4.7) AND (4.8)

	75 WSR	68 WSR		75 WSR	68 WSR
a ₀	-150.8	5.065	b_0	-279879	0
a ₁	464.0	4.355	b_1	1119110	41912
a ₂	-534.9	-33.25	b ₂	-1792230	-156380
a ₃	283.8	37.44	b ₃	1457320	225770
a_4	-67.81	-15.32	b_4	-626658	-157420
a ₅	6.008	2.143	b ₅	134280	54116
a ₆	0.04987	0.042	b_6	-12805	-8844.2
			b ₇	903.18	871.50

Gypsum slurries at 68 WSR with different foam contents show similar trends to 75 WSR slurries for both n and K as depicted in Figs. 4.6 and 4.7. The values are typically similar albeit large differences at foam contents of 0.4, 0.5 were observed for n and at foam contents of 0.2, 0.5 for K. Higher viscosities were expected at 68 WSR compared to 75 WSR since less water content versus stucco content was reduced. It is true at low shear rates less than 10 s⁻¹, but the viscosities fall more rapidly at 68 WSR as the shear rates increases, so that the viscosities of 68 WSR slurry are lower than those of 75 WSR slurry at high shear rates as shown in Fig. 4.8.



Fig. 4.6. The exponent n vs. foam content for the whole range for 68 WSR and 75 WSR slurries.



Fig. 4.7. The consistency index K vs. foam content for the whole range for 68 WSR and 75 WSR slurries.



Fig. 4.8. Comparison of viscosity vs. shear rate for 68 WSR and 75 WSR slurries with 10% v/v foam (a), and 40% v/v foam (b).

4.4.2. Shear flow of 75 WSR and 75 WSR-FI slurries

The gypsum slurries used for 75 WSR-FI had different foam contents which ranged from 0% to 70% v/v with a 10% v/v increment. When the foam content is higher than 70% v/v, the foam was unable to mix with the gypsum slurry because the corresponding reduced amount of water in the slurry itself (before foam was added, reduced to accommodate for water brought with foam and have 75 WSR fixed) results in a very thick slurry. This corresponds to slurries with less than 50 WSR in the original composition (before foam was added) as shown in TABLE 4.4. Several trials were done for each slurry composition to acquire reliable statistics. The results were fairly reproducible from 0% foam to 70% v/v foam, as shown in Fig. 4.9(b).

TABLE 4.4

MIXING GYPSUM SLURRIES OF 75 WSR-FI WITH DIFFERENT FOAM CONTENTS

Volume % Foam	Stucco	Water (g)			_		
	(g)	unfoamed water (a)	water in foam (b)	Total (a+b)	WSR	Remarks	
10	400	296	4	300	75	2 seconds of foam	
20	400	292	8	300	75	4 seconds of foam	
30	400	286	14	300	75	7 seconds of foam	
40	400	278	22	300	75	11 seconds of foam	
50	400	268	32	300	75	16 seconds of foam	
60	400	254	46	300	75	23 seconds of foam	
70	100	59	16	75	75	8 seconds of foam	
80	100	49	26	75	75	13 seconds of foam	
90	100	31	44	75	75	22 seconds of foam	



(a)



Fig. 4.9. The shear viscosity vs. shear rate for all compositions at 75 WSR (a) and 75 WSR-FI (b). The results correspond to an average of several trials.

To evaluate the rheological behavior, based on the previous results, the data for gypsum slurries with different foam content were processed using Eq. (4.1). The results on the density values and the rheological parameter values of 75 WSR-FI slurries are collected in TABLE 4.5. They show that the values of the exponent n vary from 0.16 to 0.34. The values of K vary from 514 to $191(g/cm \times s^{2-n})$. The rheological parameters for the range of foam content from 0% to 70% v/v were interpolated using polynomial curve

fitting (for both K and n). For both n and K values a 5th order polynomial was used as described in Fig. 4.10. To confirm the validity of the polynomial interpolation, error bars are included for the range of foam content investigated. It is shown that the polynomial interpolations are in good agreement with the processed n and K values though the foam contents 0.2, 03, and 0.4 are reproduced less accurately than the other contents. For the exponent n and the constistency index K, the overall trend shows that the values are rather close for different foam contents except the foam contents of 0.4 and 0.5. For example, the n values are mostly from 0.16 to 0.27 and the K values are from 406 to 514 (g/cm×s²⁻ⁿ). However, the density of different foam contents shows a large difference as shown in TABLE 4.5. The values of the coefficients of the polynomials are presented in the polynomial functions in TABLE 4.6. To have a good match between the data points and the polynomial functions, 4 digits of the coefficient were required for n and K.

TABLE 4.5

RHEOLOGICAL PARAMETERS OF GYPSUM SLURRIES FOR 75 WSR AND 75

Volume % Foam	75 WSR			75 WSR-FI		
	Density (g/cm ³)	n	K	Density	n	K
			$(g/cm \times s^{2-n})$	(g/cm^3)	11	$(g/cm \times s^{2-n})$
0	1.573	0.05	901	1.51	0.27	434
10	1.429	0.20	483	1.37	0.21	512
20	1.252	0.13	482	1.24	0.20	420
30	1.109	0.03	495	1.10	0.16	513
40	0.963	0.25	200	0.95	0.36	191
50	0.794	0.33	99	0.80	0.34	239
60	0.653	0.24	127	0.66	0.23	406
70	0.434	0.18	59	0.47	0.18	514

WSR-FI WITH DIFFERENT FOAM CONTENTS



Fig. 4.10. The polynomial curve fitting for exponent n (a) and consistency index K (b) with error bars for 75 WSR-FI slurry.

The polynomial interpolation functions with foam volume content c as a variable are listed below

$$\mathbf{n}(\mathbf{c}) = \mathbf{a}_0 \mathbf{c}^5 + \mathbf{a}_1 \mathbf{c}^4 + \mathbf{a}_2 \mathbf{c}^3 + \mathbf{a}_3 \mathbf{c}^2 + \mathbf{a}_4 \mathbf{c} + \mathbf{a}_5 \tag{4.9}$$

$$\mathbf{K}(\mathbf{c}) = \mathbf{b}_0 \mathbf{c}^5 + \mathbf{b}_1 \mathbf{c}^4 + \mathbf{b}_2 \mathbf{c}^3 + \mathbf{b}_3 \mathbf{c}^2 + \mathbf{b}_4 \mathbf{c} + \mathbf{b}_5$$
(4.10)

where the coefficients are listed in TABLE 4.6.

TABLE 4.6

THE INTERPOLATION COEFFICIENTS CORRESPOND TO EQS. (4.9) AND (4.10)

a_0	137.5	b_0	-15734
a_1	-236.0	b_1	26645
<i>a</i> ₂	134.3	b_2	-14699
<i>a</i> ₃	-27.53	b_3	28320
<i>a</i> ₄	1.247	b_4	-1310
<i>a</i> ₅	0.267	b_5	440

Gypsum slurries at 75 WSR-FI with different foam contents show a similar trend for density to that for the 75 WSR slurries as depicted in Fig. 4.11. However, the trends for the n and K values as the functions of the foam content are different as shown in Figs. 4.12 and 4.13. The values for n have large differences at foam contents of 0.4 and 0.5 and for K at foam contents of 0.6 and 0.7. Higher viscosities were expected for the 75 WSR-FI composition compared to the 75 WSR one, since water content versus stucco was reduced in the former. This was shown to be true, therefore higher foam content creates higher viscosity differences as shown in Fig. 4.14.



Fig. 4.11. Density vs. foam content for 75 WSR-FI and 75 WSR slurries.



Fig. 4.12. The exponent n vs. foam content for the whole range for 75 WSR-FI and 75 WSR slurries.



Fig. 4.13. The consistency index K vs. foam content for 75 WSR-FI and 75 WSR slurries.



Fig. 4.14. Comparison of viscosity vs. shear rate for 75 WSR-FI and 75 WSR slurries with 20% v/v foam (a), and 60% v/v foam (b).

4.4.3. Shear flow of 75 WSR-FI and 70 WSR-FI slurries

The gypsum slurries of 70 WSR-FI had different foam contents which were from the range from 0% to 60% v/v with a 20% v/v increment. When the foam content is higher than 60% v/v, the foam was unable to mix with the gypsum slurry because the corresponding reduced amount of water in the slurry itself (before foam was added, reduced to accommodate for water brought with foam and have 70 WSR fixed) results in very thick slurry. The viscosities of 70WSR-FI were compared with 75 WSR-FI to study how the amount of water changes affects the shear viscosities as shown in Fig. 4.15. The viscosities of 70 WSR-FI are larger than those of 75 WSR-FI. However, the viscosities of 70 WSR-FI decrease more rapidly with higher foam contents compare to 75 WSR-FI. The more foam content, the viscosity difference at high shear rates are smaller between 75 WSR-FI and 75 WSR as shown in Fig. 4.16.



Fig. 4.15. Comparison of viscosity vs. shear rate for 70 WSR-FI and 75 WSR-FI slurries for all foam compositions.



Fig. 4.16. Comparison of viscosity vs. shear rate for 70 WSR-FI and 75 WSR-FI slurries with different foam contents. (a) 0% foam, (b) 20% v/v foam, (c) 40% v/v foam, (d) 60% v/v foam.

To evaluate the rheological behavior, the data for gypsum slurries with different foam content were processed using Eq. (4.1). The results for the values of the rheological parameter of 70 WSR-FI slurries are combined in TABLE 4.7. They show that the values of the exponent n vary from 0.01 to 0.21. The values of K vary from 1334 to 2894 $(g/cm \times s^{2-n})$. Gypsum slurries at 70 WSR-FI with different foam contents show quite different trends compared to 75 WSR-FI slurries for both n and K as depicted in Figs. 4.17 and 4.18. The values of n rapidly decreased to 0.01 at foam contents of 0.2 and stayed at the same level up to 60v/v% foam. As for K, higher viscosities were expected for 70 WSR-FI compared to 75 WSR-FI, since less water content versus stucco inevitably results in higher viscosities. However the difference between them was reduced as the foam content increased.

TABLE 4.7

RHEOLOGICAL PARAMETERS OF GYPSUM SLURRIES FOR 70 WSR-FI AND 75 WSR-FI WITH DIFFERENT FOAM CONTENTS

Volume % Foam	70 WSR-FI			75 WSR-FI		
	Density (g/cm ³)	n	K (g/cm×s ²⁻ⁿ)	Density (g/cm ³)	n	$\frac{K}{(g/cm \times s^{2-n})}$
0	1.59	0.21	1552	1.51	0.27	434
20	1.32	0.01	2894	1.24	0.20	420
40	0.97	0.01	1541	0.95	0.36	191
60	0.70	0.01	1344	0.66	0.23	406



Fig. 4.17. The exponent n vs. foam content for the whole range for 70 WSR-FI and 75 WSR slurries.



Fig. 4.18. The consistency index K vs. foam content for 70 WSR-FI and 75 WSR-FI slurries.

4.5. Conclusion

Rheological behavior of gypsum slurries with foam generally shows shear thinning, but the thinning rates are all different with different foam contents and different WSR. The viscosity of the gypsum slurries with foam decreases as the foam content increases. However, for pure foam the viscosity is higher than the viscosity of 75 WSR corresponding to the 70% v/v or higher foam contents. It is interpreted as different physical mechanisms responsible for the rheological behavior of pure foam compared to that of gypsum slurries. The exponent n tends to increase as the foam content increases, the constancy index K tends to decrease as the foam content increases, though both dependences show fluctuations. The 6th order polynomial interpolations for n and K show a good agreement with the processed values and can be used in numerical calculations of flows of gypsum slurries with foams. The comparison of the results for 68 WSR slurries studied with those for 75 WSR slurries showed that the viscosities of 68 WSR slurries are higher at lower shear rates, but lower at the higher shear rates. Variations of the rheological parameter values n and K in 75 WSR-FI slurries with different foam contents are smaller than those found previously for 75 WSR slurries. Nevertheless, the oscillatory character of the dependences of n and K on the foam content has been confirmed. The density dependences for 75 WSR-FI and 75 WSR on foam content are almost the same. The 5th order polynomial interpolations for n and K show a good agreement with the processed values and can be used in numerical calculations of flows of 75 WSR-FI gypsum slurries with foams. The viscosities of 70 WSR-FI decrease with increasing foam content. The comparison of the results for 70 WSR-FI slurries studied with those for 75 WSR-FI slurries showed that the viscosities of 70 WSR-FI slurries decrease more rapidly than those of 75 WSR-FI as the shear rates increase.

5. FOAM CONSOLIDATION AND DRAINAGE

5.1. Introduction

In the present work, free drainage of foam in an axisymmetric cylindrical gravity settler was investigated and a new theory involved in foam drainage rate in the framework of consolidation theory was developed. Then the predictions the theory made are compared to the experiment results.

5.2. Theoretical Background

5.2.1. General equations

According to Kornev et al. (1999), foam in which gas bubbles have already crowded, begun to collide, and press on each other significantly, possess elasticity. The bubbles and surfactant-stabilized liquid lamellae in strong foam form an elastic skeleton. In addition, solvent can drain through the lamellae driven by a pressure gradient according to Darcy's law

$$\mathbf{v}^{\mathrm{LS}} = \mathbf{v}^{\mathrm{L}} - \mathbf{v}^{\mathrm{S}} = -\frac{k}{\mu} \nabla p \tag{5.1}$$

where \mathbf{v}^{L} , \mathbf{v}^{S} and \mathbf{v}^{LS} are the liquid and skeleton velocities and the liquid-relative-toskeleton average velocity, respectively, k is the skeleton permeability on the order of $k=\delta^{2}/12$ with δ being the average lamella thickness, μ is the solvent viscosity and p is the solvent pressure.
Foam, as a system, comprised of an elastic skeleton assumed to follow Hooke's law with viscous liquid drainage in the lamellae perfectly resembles the medium dealt with by consolidation theory of soil mechanics (Terzaghi, 1947; Fowler, 1997) and is a medium kindred of liquid-saturated concentrated slurries (Yarin et al., 2006). In such cases, a concerted volumetric deformation of the elastic skeleton comes in reaction to the viscous drainage of an incompressible viscous liquid, which is described by the balance equation

$$\frac{\partial \varepsilon_{v}}{\partial t} - \frac{k}{\mu} \nabla^{2} p = 0$$
(5.2)

following from Darcy's law Eq. (5.1) with t being time.

In Eq. (5.2) the volumetric strain is given by

$$\varepsilon_{v} = \operatorname{div} \mathbf{u}$$
 (5.3)

where \mathbf{u} is the displacement vector, such that

$$\mathbf{v}^{\mathrm{S}} = \frac{\partial \mathbf{u}}{\partial t} \tag{5.4}$$

The consolidation theory, according to Terzaghi's principle (Fowler, 1997), implies that the total stress σ in any cross-section of the system under consideration is comprised of the isotropic part solely associated with the liquid pressure p and the deviatoric elastic part σ' , where σ' is the effective stress tensor in the skeleton responsible for its deformation. In other words, the total stress tensor σ =-p**I**+ σ ' where **I** is tensor unit. Since deformations are assumed to be relatively slow, the momentum balance equation reduces to the following form (Fowler, 1997)

$$\nabla \cdot \boldsymbol{\sigma} - \nabla \mathbf{p} = 0 \tag{5.5}$$

The deviatoric stress tensor is given by Hooke's law (Landau et al., 1970)

$$\mathbf{\sigma}' = \frac{E}{1+\nu} \mathbf{\varepsilon} + \mathbf{I} \frac{E\nu}{(1+\nu)(1-2\nu)} \operatorname{div} \mathbf{u}$$
(5.6)

where $\mathbf{\epsilon} = (1/2) [\nabla \mathbf{u} + \nabla \mathbf{u}^{T}]$ is the strain tensor, E is the effective Young's modulus and v is the effective Poisson's ratio (both assumed to be constant) of the elastic skeleton.

We assume that the average bubble radius a in the foam is sufficiently large to neglect capillary pressure in the bubbles $4\gamma/a$ (with γ being the surface tension, and the factor 4 corresponds to the two free surfaces surrounding a bubble) compared to the surrounding atmospheric pressure p_0 , which is indeed true for the bubbles with $a \approx 300 \,\mu\text{m}$ characteristic of our foams. Therefore, gas inside the bubbles has pressure p_0 . Foam elasticity results from the compressibility of the gas inside the bubbles since liquid is practically incompressible. Assuming gas inside the bubbles being non-condensable and ideal, one can easily see that the bulk modulus of foam K is equal to the gas pressure p_0 , i.e. K=E/[3(1-2v)]= p_0 . Therefore,

$$\mathbf{E} = 3\mathbf{p}_0\boldsymbol{\varphi}, \quad \mathbf{v} = \frac{1-\boldsymbol{\varphi}}{2} \tag{5.7}$$

where φ is the gas volume fraction in the initial foam. It is emphasized that Eq. (5.7) relates the foams effective Young's modulus and Poisson's ratio, E and v, to the known parameters p_0 and φ . The second Eq. (5.7) shows that as the gas volume fraction in the initial foam $\varphi \rightarrow 0$, the foam becomes incompressible, since correspondingly $\nu \rightarrow 1/2$. Equation (5.6) allows one to find, as in (Landau et al., 1970), that

$$\boldsymbol{\nabla} \cdot \boldsymbol{\sigma} = \frac{1}{2} \frac{E}{(1+\nu)} \left[\boldsymbol{\nabla}^2 \boldsymbol{u} + \frac{1}{1-2\nu} \boldsymbol{\nabla} (\text{div}\boldsymbol{u}) \right]$$
(5.8)

Equations (5.2) and (5.3) and combined Eqs. (5.5) and (5.8) form a system of two equations for the two unknowns p and \mathbf{u} , namely

$$\frac{\partial (\operatorname{div} \mathbf{u})}{\partial t} = \frac{k}{\mu} \nabla^2 p \tag{5.9}$$

$$\frac{1}{2} \frac{\mathrm{E}}{(1+\nu)} \left[\nabla^2 \mathbf{u} + \frac{1}{1-2\nu} \nabla(\mathrm{div}\mathbf{u}) \right] = \nabla p \tag{5.10}$$

Taking the divergence of Eq. (5.10) yields

$$\frac{\mathrm{E}(1-\nu)}{(1+\nu)(1-2\nu)}\nabla^{2}(\mathrm{div}\mathbf{u}) = \nabla^{2}p$$
(5.11)

Equation (5.9) allows us to exclude p from Eq. (5.11), which yields the following scalar equation containing solely **u**, in particular, ε_v =div**u**,

$$\frac{\partial \varepsilon_{v}}{\partial t} = c_{v} \nabla^{2} \varepsilon_{v}$$
(5.12)

where

$$c_{v} = \frac{k}{\mu} \frac{E(1-v)}{(1+v)(1-2v)}$$
(5.13)

Equation (5.12) describes the evolution of the volumetric strain during skeleton consolidation accompanying liquid drainage. It coincides formally with the standard parabolic heat transfer equation, where the role of the transport coefficient, in the present case, is played by the consolidation coefficient c_v given by Eq. (5.13).

In addition, Eq. (5.11) can be integrated, which yields the following relation between p and $\epsilon_{\rm v}$

$$p = \frac{E(1-v)}{(1+v)(1-2v)}\varepsilon_v + F(\mathbf{r}, t)$$
(5.14)

where F is any harmonic function of the position vector **r**, which can also contain time as a parameter.

5.2.2. Gravity settler

In an axisymmetric cylindrical gravity settler initially filled with uniform foam up to height h a new physical factor, namely gravity, plays role and liquid drainage to the bottom is anticipated (Fig. 5.1).



Fig. 5.1. A filled cylinder of foam with surfactant solution (drained liquid) below. The top of the foam is at z=0 and the bottom of the cylinder is at z=-h.

In presence of gravity, Darcy's law (5.1) is amended and takes the following form (Barenblatt et al., 1990)

$$\mathbf{v}^{LS} = \mathbf{v}^{L} - \mathbf{v}^{S} = -\frac{k}{\mu} \nabla \left(\mathbf{p} + \rho g z \right)$$
(5.15)

where ρ is the liquid density, g gravity acceleration, and z is the vertical coordinate with z=0 corresponding to the free surface and z=-h to the bottom.

Equation (5.15) expresses the fact that the hydrostatic pressure distribution in lamellae at rest (p=p₀-pgz with z being negative, should not result in any drainage against gravity, as is physically expected; p₀ is the atmospheric pressure). The presence on the gravity-related term in Eq. (5.15) does not preclude arriving at Eq. (5.2) by the previous derivation, since $\nabla^2 z \equiv 0$. Also, Eq. (5.5) which expresses the balance of the elastic stresses due to the surfactant lamellae and liquid pressure (whatever it is) acting on them does not change. Then Eqs. (5.8)-(5.14) also do not change. In particular, Eqs. (5.12) and (5.14) in the present case reduce to the following equations

$$\frac{\partial \varepsilon_{v}}{\partial t} = c_{v} \frac{\partial^{2} \varepsilon_{v}}{\partial z^{2}}$$
(5.16)

$$p = \frac{E(1-v)}{(1+v)(1-2v)} \varepsilon_v + Az + B$$
(5.17)

where A and B are constants.

Substituting Eq. (5.17) into Eq. (5.16), we obtain the following equation for the liquid pressure

$$\frac{\partial p}{\partial t} = c_v \frac{\partial^2 p}{\partial z^2}$$
(5.18)

The drainage velocity v in the gravity direction according to Eq. (5.15) is given by

$$\mathbf{v} = -\frac{\mathbf{k}}{\mu} \frac{\partial (\mathbf{p} + \rho \mathbf{g} \mathbf{z})}{\partial \mathbf{z}}$$
(5.19)

The drainage velocity u is equal to zero at the bottom (at z=-h). It can also be assumed to equal zero at the free surface, so that the boundary conditions for Eq. (5.18) at t>0 read

$$\left. \frac{\partial \mathbf{p}}{\partial \mathbf{z}} \right|_{\mathbf{z}=0 \text{ or } \mathbf{z}=-\mathbf{h}} = -\rho g \tag{5.20}$$

The boundary condition (5.20) at z=0 implies that foam is pinned at the column top at z=0, which is indeed the case as discussed below. It is also emphasized that in the experiments, the drained (un-foamed) liquid column accumulates near the settler bottom in the course of drainage and the lower boundary of foam moves from z=-h up. However, during the entire drainage process the height of this drained liquid column is less than 7 cm, whereas the foam column height is 104 cm. Therefore, in the first approximation, dealing with foams with the initial gas volume fraction φ of about 0.95, we neglect the effect of motion of the lower foam boundary and displace the boundary condition (5.20) to z=-h for any t.

The initial condition corresponds to uniform foam

$$p|_{t=0} = p_0$$
 (5.21)

Equations (5.18) and (5.20) show that the problem has an integral invariant

$$\int_{-h}^{0} pdz = p_0 h = \text{const}$$
(5.22)

which can be recast with the help of Eq. (5.17) into

$$\int_{-h}^{0} \varepsilon_{v} dz = \frac{(1+v)(1-2v)}{E(1-v)} \left(p_{0}h + \frac{Ah^{2}}{2} - Bh \right) = \text{const}$$
(5.23)

It is easy to see that the left hand side of Eq. (5.23) corresponds to nothing but the liquid content in the foam layer $-h \le z \le 0$, which obviously should not change in time. Indeed, according to Eq. (5.4) it does not.

It is convenient to introduce $P=p+\rho gz$, and render t dimensionless by h^2/c_v ; p, p₀ and P by ρgh ; and z by h. Then, the problem (5.18), (5.20) and (5.21) can be recast to the following dimensionless form

$$\frac{\partial \mathbf{P}}{\partial t} = \frac{\partial^2 \mathbf{P}}{\partial z^2} \tag{5.24}$$

$$\left. \frac{\partial \mathbf{P}}{\partial z} \right|_{z=0 \text{ or } z=-1} = 0 \qquad \text{at } t>0 \tag{5.25}$$

$$P = P_0(z) = p_0 + z$$
 at t=0 (5.26)

where bars over the dimensionless parameters are omitted for brevity.

The integral invariant (5.22) takes the form

$$\int_{-1}^{0} Pdz = p_0 - \frac{1}{2}$$
(5.27)

corresponding to Eqs. (5.24)-(5.26).

Finding the solution of the problem (5.24)-(5.26) and returning from P to the dimensionless pressure p, one obtains

$$p = p_0 - \sum_{n_{odd}=1}^{\infty} \frac{4}{\pi^2 n^2} \Big[1 - \exp(-\pi^2 n^2 t) \Big] \cos(\pi n z)$$
(5.28)

It is easy to see that as $t \to \infty$, according to Eq. (5.28) $p \to p_{\infty} = p_0 - z - 1/2$. In the dimensional units it means that pressure distribution tends to

$$p = p_0 - \frac{\rho g h}{2} - \rho g z \tag{5.29}$$

As a result, one expects that at the pinned free surface z=0 (i.e. the free surface of foam which cannot slip down the settler wall during foam drainage), pressure will become lower than the atmospheric pressure, namely, p_0 - $\rho gh/2$. The difference is small,

 $\rho gh/2 \approx 0.01 p_0$, and represents itself the price of the assumption that the free surface is pinned. In reality, the free surface was pinned at the settler wall and cap.

Using the solution (5.28) and Eq. (5.19), we find the dimensional drainage velocity in the following form (albeit z is still dimensionless)

$$v = -\frac{k\rho g}{\mu} \left\{ \sum_{n_{odd}=1}^{\infty} \frac{4}{\pi n} \left[1 - \exp(-\pi^2 n^2 t) \right] \sin(\pi n z) + 1 \right\}$$
(5.30)

Equation (5.30) shows that at t=0 at any z (in particular at the settler bottom) the drainage velocity v=-k $\rho g/\mu$. At time t>h²/($\pi^2 c_v$), the drainage velocity at any z doubles, v=-2k $\rho g/\mu$.

Foam in the settler does not detach from the walls. Therefore, in the present case $u_{xx}=u_{yy}=0$, while $\varepsilon_v=u_{zz}$. Then, according to the Eq. (5.6), we find in the present case

$$\sigma_{zz} = \frac{E\epsilon_{v}(1-v)}{(1+v)(1-2v)} - p$$
(5.31)

whereas the lateral stresses σ_{xx} and σ_{yy} are non-zero, as is expected in the laterally restricted situation.

Using Eq. (5.17), it is easy to see that

$$\varepsilon_{v} = \frac{(1+v)(1-2v)}{(E/\rho gh)(1-v)} \sum_{n_{odd}=1}^{\infty} \frac{4}{\pi^{2}n^{2}} \exp(-\pi^{2}n^{2}t) \cos(\pi nz)$$
(5.32)

with t and z being dimensionless.

Then, from Eqs. (5.31), (5.32) and (5.28) we find that the stress, rendered dimensionless by ρgh , is given by

$$\sigma_{zz} = -p_0 - \sum_{n_{odd}=1}^{\infty} \frac{4}{\pi^2 n^2} \Big[2 - \exp(-\pi^2 n^2 t) \Big] \cos(\pi nz)$$
(5.33)

This equation shows that the stress on the bubbles near the bottom is always compressive (negative) and its magnitude increases in time from the dimensionless value of $(p_0+1/2)$ to (p_0+1) . The experiments show that the increasing pressure near the settler bottom leads to breakup of larger bubbles and formation of the smaller ones. This, however, cannot change the effective Young's modulus E and Poisson's ration v of the foam if no gas is lost, i.e. φ =const [cf. Eqs. (5.7)].

It is emphasized that the above theory assumes that the lamellae thicknesses in the foam are sufficiently large to neglect the effect of the long-range forces (e.g. the van der Waals forces) associated with the disjoining pressure (Derjaguin, 1934; Kornev et al., 1999). This assumption is sufficiently accurate for the lamellae thicknesses above 1 μ m. However, at the later stage of liquid drainage from the foam the lamellae can thicken below this threshold and the disjoining pressure tends to stabilize them, which decelerates the latest stage of the drainage process (Kornev et al., 1999). This effect will also be visible in the experimental results described in section 5.3, and the present theory cannot be applied to the latest stage of drainage.

5.3. Results and Discussion

The evolution of the drainage rate measured in the work of another PhD student D. Pelot is used for comparison with the theoretical predictions of section 5.2. When drainage begins, its initial rate is relatively slow and is approximately constant (Fig. 5.2). Next, the drainage rate increases in time until a maximum is reached, corresponding to twice the initial drainage rate, and proceeds at this constant higher rate (Fig. 5.2). Fig. 5.2 also shows that the drainage process begins to slow down compared to the maximum rate of drainage at t>800 s. This phenomenon is probably characteristic of older and/or dryer foam, in which the lamellae thickness is below 1 μ m and the disjoining pressure (not accounted for in the present theory) decelerates the drainage process.

The quantitative parameters of the data shown in Fig. 5.2 are listed in TABLE 5.1. In particular, it includes the minimum and maximum slopes, and the ratio of the maximum to the minimum slopes. It is emphasized that these slopes are directly proportional to the drainage rates, i.e. to the drainage rate v predicted by Eq. (5.30). It is clear from TABLE 5.1 that increasing shampoo concentrations reduce both minimum and maximum rates of drainage. However, their ratios are approximately the same in all cases and are close to 2, which agree with the theoretical prediction of section 5.2. This reveals that the theory presented in section 5.2 is capable of describing foams.



Fig. 5.2. Height of liquid column drained for the first 1000 s (before the drainage process cessation is felt) in Trial 1 of 2 %v/v shampoo. The two lines show the crossover of a slow (initial) and fast (maximum) drainage processes. The ratio of maximum drainage rate to initial drainage rate in this case is 2.03. The experimental data of D. Pelot.

TABLE 5.1

RESULTS FOR DIFFERENT CONCENTRATIONS OF SHAMPOO. THE

Concentration	2 %v/v Shampoo		4 %v/v Shampoo		8 %v/v Shampoo	
Trial number	1	2	1	2	1	2
Viscosity (cP)	1.0	1.0	1.0	1.0	1.1	1.1
Minimum slope $x10^3$ (cm ³ /s)	15.0	14.6	9.4	11.3	9.1	9.8
Maximum slope x10 ³ (cm/s)	30.5	29.3	20.9	23.0	21.2	21.3
Max. slope/Min. slope	2.03	2.01	2.22	2.04	2.33	2.17

EXPERIMENTAL DATA OF D. PELOT

5.4. Conclusion

The present theory treats foam as a consolidating medium possessing elastic properties predominantly due to gas present in the bubbles, as well as a Darcy-like drainage of liquid from their interconnected lamellae. It is predicted that the drainage process begins with a slow stage. This stage lasts on the scale of the characteristic consolidation time. After that it is replaced by a faster drainage stage in which the rate of drainage doubles. This prediction was confirmed by the current experiments with foams with different surfactant concentrations and a polymer additive. At the latest stage of foam existence, the drainage could be attributed to a deceleration by the disjoining pressure in liquid lamellae.

6. FOAM THREADS: THEORY AND EXPERIMENT

6.1. Introduction

In this Chapter, a Pantene shampoo was used as a surfactant to prepare foam. In some cases, a water-soluble polymer, Polyethylene oxide (PEO), was added. Foam thread shape was predicted by consolidation theory (section 6.2) and compared to our experiments on foam stretching described in section 6.3. The result and discussion is given in section 6.4. Conclusions are drawn in section 6.5.

6.2. Theoretical Background

Consider an axisymmetric foam thread with x being its axial coordinate and y and z any other orthogonal axes (x=0 at the thread center), cf. Fig. 6.1.



Fig. 6.1. Sketch of a foam thread.

Assume a quasi-one-dimensional approximation being valid in the thread, which is a plausible assumptions for free liquids jets and threads in such situations (Yarin, 1993). Then, Eq. (5.12) reduces to the following form

$$\frac{\partial \varepsilon_{v}}{\partial t} = c_{v} \frac{\partial^{2} \varepsilon_{v}}{\partial x^{2}}$$
(6.1)

The axial total stress is given by

$$\sigma_{xx} = \frac{E}{1+\nu} u_{xx} + \frac{E\nu}{(1+\nu)(1-2\nu)} \varepsilon_{\nu} - p$$
(6.2)

whereas both radial total stresses in a thin thread are equal to the local capillary pressure $p_{\sigma}(x,t)$ times -1,

$$\sigma_{yy} = \frac{E}{1+\nu} u_{yy} + \frac{E\nu}{(1+\nu)(1-2\nu)} \varepsilon_{\nu} - p = -p_{\sigma}$$
(6.3)

$$\sigma_{zz} = \frac{E}{1+\nu} u_{zz} + \frac{E\nu}{(1+\nu)(1-2\nu)} \varepsilon_{\nu} - p = -p_{\sigma}$$
(6.4)

The capillary pressure in the thread p_{σ} is related to the cross-sectional radius of the thread a(x,t) via the Young-Laplace equation which takes the following form in the present case

$$p_{\sigma} = \sigma \left\{ \frac{1}{a \left[1 + (da/dx)^{2} \right]^{1/2}} - \frac{d^{2}a/dx^{2}}{\left[1 + (da/dx)^{2} \right]^{3/2}} \right\}$$
(6.5)

where σ is the surface tension coefficient.

Equations (6.3) and (6.4) yield

$$u_{yy} = u_{zz} = \frac{(1+\nu)(p-p_{\sigma})}{E} - \frac{\nu \varepsilon_{\nu}}{(1-2\nu)}$$
(6.6)

By definition, $u_{xx}+u_{yy}+u_{zz}=\varepsilon_v$. Using the latter expression together with Eq. (6.6), we find

$$u_{xx} = \frac{\varepsilon_{v}}{(1-2v)} - \frac{2(1+v)(p-p_{\sigma})}{E}$$
(6.7)

Substituting Eq. (6.7) into Eq. (6.2), we find

$$\sigma_{xx} = \frac{E\varepsilon_{v}}{1-2v} - 3p + 2p_{\sigma}$$
(6.8)

In the one-dimensional case F in Eq. (5.14) can be only a constant F_0 . Then, excluding pressure p from Eq. (6.8) with the help of Eq. (6.5) yields

$$\sigma_{xx} = -\frac{2E\varepsilon_{v}}{1+v} - 3F_{0} + 2p_{\sigma}$$
(6.9)

In the present one-dimensional case the axial projection of Eq. (5.5) reduces to $\partial \sigma_{xx} / \partial x = 0$, and thus, $\sigma_{xx} = \sigma_{xx\ell}$ where $\sigma_{xx\ell}$ is the stress acting at the thread edge at $x = \ell$. As a result, Eq. (6.9) takes the following form of the relation between the volumetric strain ε_v and the capillary pressure in the thread p_{σ}

$$\varepsilon_{v} = \frac{(1+v)}{E} p_{\sigma} - \text{const}$$
(6.10)

where const = $(1 + v)(3F_0 + \sigma_{xx\ell})/2E$.

Substituting Eq. (6.10) into Eq. (6.1) yields the following dimensionless problem for the capillary pressure

$$\frac{\partial \mathbf{p}_{\sigma}}{\partial t} = \frac{\partial^2 \mathbf{p}_{\sigma}}{\partial x^2} \tag{6.11}$$

$$\mathbf{t} = \mathbf{0}, \quad \mathbf{p}_{\sigma} = \mathbf{p}_{\sigma 0}(\mathbf{x}) \tag{6.12}$$

$$x = 0, \quad \frac{\partial p_{\sigma}}{\partial x} = 0 \tag{6.13}$$

$$x = 1, p_{\sigma} = 0$$
 (6.14)

In Eqs. (6.11)-(6.14) ℓ is taken as the scale of the coordinate, ℓ^{-1} as the scale of the capillary pressure and ℓ^2/c_v as the scale of time, while bars over the dimensionless parameters are omitted for brevity. Equation (6.12) poses the initial condition as the capillary pressure distribution $p_{\sigma 0}(x)$ corresponding to any outcome of the initial prestetching of the filament in the experimental apparatus described in the previous section. Equations (6.13) and (6.14) impose the boundary conditions at the thread center (symmetry) and the edge, respectively. The latter boundary condition implies that the thread is attached to an edge area fully released from the capillary pressure which mimics the experimental situation in the edge areas where the thread is attached to the solid disks. This means that at the edge areas the thread acquires a catenoidal shape and capillary pressure vanishes.

According to Eqs. (6.11)-(6.14), the evolution of any initial shape of the thread corresponding to an arbitrary initial distribution $p_{\sigma 0}(x)$ inevitably leads to catenoidal shape with $p_{\sigma}(x)$ approaching zero over the whole thread. Indeed, the solution of Equations (6.11)-(6.14) reads

$$p_{\sigma}(x,t) = \sum_{n=0}^{\infty} M_n \exp\left[-\pi^2 (n+1/2)^2 t\right] \cos \pi (n+1/2) x$$
(6.15)

$$M_{n} = 2 \int_{0}^{1} p_{\sigma 0}(x) \cos \pi (n + 1/2) x dx$$
 (6.16)

The solution (6.15) and (6.16) shows that on the scale of t=1, i.e. on the scale of the dimensional time $t \approx \ell^2 / c_v$ the capillary pressure $p_{\sigma}(x,t)$ becomes zero, which corresponds according to Eq. (6.5) to the following dimensionless catenoidal shape of the thread

$$a = C_1 \cosh(x/C_1 + C_2)$$
(6.17)

where C_1 and C_2 are the integration constants and a and x are rendered dimensionless by ℓ . Note, that if needed, the whole history of the thread evolution a (x,t) from the initial shape to the terminal catenoidal shape (6.17) can be easily reproduced by substituting Eqs. (6.15) and (6.17) into Eq. (6.5) (which should be rendered dimensionless) and solving the latter as an ODE for a.

The final fate of the thread is quite peculiar. Indeed, Eq. (5.3) should satisfy two boundary conditions, namely

$$x = 0, \quad \frac{da}{dx} = 0; \quad x = 1, \quad a = \overline{a_1} = \frac{a_1}{\ell}$$
 (6.18)

where a_1 is the dimensional value of the disk radius.

The boundary condition (6.18) at x=0 means that C₂=0. Also, it is easy to see that $\ell > 0.662a_1$ the catenoidal shape (6.17) cannot satisfy the boundary conditions (6.18), and thus the thread should break before the terminal configuration is attained (probably into several blobs). The semi-length $\ell = 0.662a_1$ yields a critical situation in which a single

catenoid can span the boundary conditions (6.18) which means that the foam thread will not break up but rather stabilize itself at this terminal shape, if it is stable. On the other hand, the relatively short threads with $\ell < 0.662a_1$ can stabilize themselves in the terminal catenoidal shape, but they have a choice. Indeed, at $\ell < 0.662a_1$ two different catenoids (6.17) satisfy the boundary conditions (6.18).



Fig. 6.2. Two solutions arise from Eq. (6.17) if $\ell < 0.662a_1$ which correspond to two values of $C_1 - C_1$ and $C_1^{"}$.

They might be both stable, and then the choice of the final shape depends on the initial condition (6.12) which might lead to one or another catenoid. On the other hand, one of the catenoids can be unstable (e.g. the thinner one), or both might be unstable.

6.3. Experimental

To create foam, DI water and a Classic Pantene Pro-V shampoo were mixed in a stainless steel mixing bowl. The amount of Pantene used was 2 ml, 4 ml or 8 ml out of 100 ml solution as shown in TABLE 6.1. In addition, a sample of the 8% solution was also mixed with 0.05 g of Polyethylene oxide (PEO, M_w =8 MDa) to prepare the fourth foam listed in TABLE 6.1. Using a Chefmate hand mixer with speed setting of six which is about 1,000 rpm, soap solutions were mixed for 2 min. for all cases except the foam containing PEO, for which the mixing time was 5 min. The reason that a longer mixing time was required for the foam with PEO is that initially large bubbles were present because PEO stabilizes bubbles. To break up the large bubbles and prepare a more homogeneous foam, a longer stirring time was helpful. An as-prepared foam was extracted using a 5 ml syringe and deposited into a 3 ml glass cup. Three trials with each composition were done. All foams were prepared anew for each trial.

For the experiments with foam threads, the elongational rheometer of Refs. (Yarin et al., 2004; Tiwari et al., 2009) shown schematically in Fig. 6.3a, was modified as follows. The lower support was made as a small cup which was filled with foam. The upper support was dipped into the cup and then withdrawn to make instantaneously a foam thread as depicted in Fig. 6.3b. In particular, the foam threads with the lengths of 3 mm, 4 mm, 5 mm and 6 mm were created this way. It is emphasized that after a thread has been created, its length 2ℓ did not change in the experiments, whereas the thread minimum diameter (Fig. 6.4) was diminishing under the action of surface tension. The self-thinning of a foam thread was recorded using a high speed CCD camera (RedLake

Motion) at a frame rate of 25 fps. The images were transformed to PC. The acquired data was then processed in an automated mode with a MATLAB-based image processing code.

TABLE 6.1

PARAMETERS CHARACTERIZING FOAM PREPERATION

Pantene foam concentration	Water (ml)	Pantene (ml)	PEO (g)	Mixing time (s)
2%	98	2	-	120
4%	96	4	-	120
8%	92	8	-	120
8% + 0.05g PEO	92	8	0.05	300



Fig. 6.3. (a) Schematic of the elongational rheometer (Yarin et al., 2004; Tiwari et al., 2009). (b) Making the initial foam thread.



Fig. 6.4. Image of a thread of Pantene foam between two supports of the elongational rheometer.

6.4. Results and Discussion

6.4.1. 2% Pantene concentration

The longer was the thread length 2ℓ , the smaller was the initial minimum thread diameter. The corresponding data are presented in Figs. 6.5-6.8. In all these cases the minimum thread diameter decreases in time. However, the longest-living threads are those with the shortest length. The thread shape changed due to the foam drainage under the action of the surface tension, bubble bursting, as well as due to two unwanted effects: water evaporation and perturbations introduced by the surrounding air. It is emphasized that the difference between the trials 1-3 in each of Figs. 6.5-6.8 is due to the fact that in each trial the contact line of the initial thread acquires another position, even though all the other parameters were unchanged up to the possible experimental accuracy. It means that the difference between the trials in each of the Figs. is due to the initially different threads.



Fig. 6.5. Thread thinning of 2% Pantene thread which is 3 mm long for each of the three trials. The dashed loops encircle the quasi-steady state plateaus used for analysis. The numbers near the plateaus denote the plateaus.



Fig. 6.6. Thread thinning of 2% Pantene thread which is 4 mm long for each of the three trials. The dashed loops encircle the quasi-steady state plateaus used for analysis. The numbers near the plateaus denote the plateaus.



Fig. 6.7. Thread thinning of 2% Pantene thread which is 5 mm long for each trial. The dashed circle represents the maximum plateau for each trial.



Fig. 6.8. Thread thinning of 2% Pantene thread which is 6 mm long for each trial. The dashed circle represents the maximum plateau for each trial.

The data in Figs. 6.5-6.8 show the presence of several intermediate plateaus which last about not more than 3-4 s for the 3 mm tread, where a steady state configuration has been approximately achieved. The thread shapes corresponding to these plateaus were used for comparison with the theoretical predictions of section 6.2. It is emphasized that the theory expects that a steady-state plateau should exist indefinitely. The experiments, however, show that such plateaus are only intermediate and the corresponding quasisteady states are ultimately disrupted by water evaporation and bubble bursting. For the largest plateau of each trial, the value of the corresponding thread radius in contact with the support a_1 was measured from the images similar to that in Fig. 6.4. Then, the value of ℓ/a_1 was calculated. The corresponding values of ℓ/a_1 are presented in TABLE 6.2. It is seen that only in the case of the 3 mm thread are the corresponding values of ℓ/a_1

lower than the critical value of 0.662 predicted by the theory in section 6.2. Therefore, only these plateaus can belong to the category of those plateaus for which the theory predicts the existence of the steady state. This conclusion is corroborated by the fact that the durations of the stable threads corresponding to plateaus 1-3 are much longer than for those corresponding to plateaus 4-12 (cf. TABLE 6.2). Therefore, the other plateaus corresponding to longer threads in Figs. 6.6-6.8 listed in TABLE 6.2 are just transients and cannot be real steady-state plateaus. Therefore, these longer threads at such low Pantene concentration did not approach any steady state, as expected from the theory.

The theory predicts for each value of $\ell/a_1 < 0.662$ corresponding to the plateaus of Fig. 6.5 the existence of two steady shapes of the thread. It is instructive to check whether any one of them agrees with the experimental data. If that is the case, the other one is either unrealizable due to the initial conditions at the beginning of the transformation to the steady-state or unstable.

TABLE 6.2

THE VALUES OF $\ell\,/\,a_1$ corresponding to the plateaus 1-12 in Figs. 6.5-

Plateau No.	Thread length (mm)	ℓ /a ₁	Duration of stable thread (s)
1	3	0.31	4.2
2	3	0.31	3.7
3	3	0.42	3.0
4	4	0.73	1.7
5	4	0.69	1.2
6	4	0.60	0.8
7	5	0.72	0.7
8	5	0.68	0.4
9	5	0.62	1.0
10	6	0.83	0.8
11	6	1.01	0.8
12	6	0.83	0.4

6.8.

An example of the comparison of the theoretical steady-state shapes with the steady-state experimental data from Fig. 6.5 is shown in Fig. 6.9. The case depicted there corresponds to the plateau 3, with $\ell/a_1 = 0.42$, and the two steady-state solutions correspond to $C_1 = 2.125$ and $C_1 = 0.415$. TABLE 6.3 presents the values of C_1 and C_1 for all the quasi-steady plateaus in Fig. 6.5. In all cases the steady-state solution which was

the closest to the experimental data is the one corresponding to C_1 . TABLE 6.3, as well as Fig. 6.9, also lists the maximum difference between the experimental data and the theoretical steady-state solution corresponding to the thread "bottleneck". It is emphasized that in the experiments the threads were not perfectly axisymmetric, and only the right-hand side generatrix was used for such comparisons. The right- and left-hand sides of the experimentally observed threads had different a_1 values due to some uncontrolled wettability differences in the support material and texture, which corresponded to different a_1 / ℓ values and thus to different steady-state profiles. The theoretical predictions for the both sides are compared to the experimental data in Fig. 6.10. Still, in the comparisons with both right- and left-hand side experimental profiles, the solution corresponding to C_1 was the closest to the experimental data, which hints that it was chosen by the initial conditions, an initially wider thread, in the experiments. The maximum difference (%) in TABLE 6.3 means the maximum difference value (cf. Fig. 6.9) divided by the minimum ("bottleneck") thread radius.



Fig. 6.9. Comparison between the experimental and theoretical steady-state thread shapes. 2% Pantene thread, plateau 3: 1-experimental data; 2-steady-state solution (6.17) with $C'_1 = 2.125$; 3- steady-state solution (6.17) with $C'_1 = 0.415$. Only one quarter of the thread is shown.

TABLE 6.3

THE VALUES OF C_1 AND C_1 CORRESPONDING TO DIFFERENT QUASI-STEADY STATE PLATEAUS IN FIG. 6.5, AND THE MAXIMUM DIFFERENCE (%) WITH THE RIGHT-HAND SIDE OF THE EXPERIMENTAL THREAD. 2% PANTENE CONCENTRATION WITH 3 MM THREAD LENGTH

Plateau No.	ℓ /a ₁	C_1	$C_1^{"}$	Max. diff. (%)
1	0.31	3.025	0.345	16.7
2	0.31	3.025	0.345	11.9
3	0.42	2.125	0.415	25.1



Fig. 6.10. Foam thread with the 2% Pantene concentration and 3 mm length corresponding to plateaus from Fig. 6.5: (a) plateau No. 1, (b) plateau No. 2, and (c) plateau No. 3.

6.4.2. 4% Pantene concentration

A higher Pantene concentration in the foam resulted in longer-lived threads (cf. Fig. 6.11 with Figs 6.5-6.8). Still, the foam was not perfectly stable, bubbles bursted, and there were some perturbations in the surrounding air. For 4% Pantene concentration with 3 mm thread length, the largest plateau, which lasted for 6-8 s, was found and compared to the theoretical results.



Fig. 6.11. The evolution of the 4% Pantene thread with 3 mm thread length.

The details on the comparison of the steady-state theoretical profiles with the experimental data for the 4% Pantene foams are given in TABLE 6.4 and Fig. 6.12. Once again, the theoretical solution corresponding to C'_1 was the closest to the experimental data. The maximum difference in TABLE 6.4 is approximately 20-30%. It is emphasized

that for the 4% Pantene foams the values of $\ell/a_1 < 0.662$ in TABLE 6.4 show that all the plateaus can be characterized as quasi-steady state plateaus. Note also, that their duration is significantly longer than those of the stable plateaus 1-3 in TABLE 6.2.

TABLE 6.4

THE VALUES OF C₁['] AND C₁['] CORRESPONDING TO DIFFERENT QUASI-STEADY STATE PLATEAUS IN FIG. 6.11, AND THE MAXIMUM DIFFERENCE (%) WITH THE RIGHT-HAND SIDE OF THE EXPERIMENTAL THREAD. 4% PANTENE CONCENTRATION WITH 3 MM THREAD LENGTH

Plateau No.	ℓ / a_1	C_1	$C_1^{"}$	Max. diff. (%)	Duration of stable thread (s)
13	0.60	1.255	0.585	16.7	6.2
14	0.47	1.865	0.445	25.1	8.2
15	0.25	3.855	0.305	31.3	6.2



Fig. 6.12. Foam thread with the 4% Pantene concentration and 3 mm length corresponding to different plateaus from Fig. 6.11: (a) plateau No. 13, (b) plateau No. 14 and (c) plateau No. 15.

6.4.3. 8% Pantene concentration

To make more stable foam, 8% Pantene concentration was used. It was found, indeed, that the 8% Pantene foam is more stable than the 2% and 4% Pantene foams, as it follows from the evolution of the minimum thread diameter depicted in Fig. 6.13. The
reduction of the minimum thread radius over time in Fig. 6.13 for the 8% Pantene foam is much slower than that depicted in Figs. 6.5-6.8 for the 2% Pantene foam and Fig. 6.11 for the 4% Pantene foam. The comparison of the experimental data with the theory is illustrated by TABLE 6.5 and Fig. 6.14. In this case the theoretical predictions are rather close to the experimental data and the maximum differences are just 3-5% for trials 1 and 2, though the maximum difference is about 11% for trial 3. As for the 2% and 4% Pantene foams, in the case of the 8% Pantene foam the theoretical solution corresponding to C₁['] was the closest to the experimental data. It is emphasized that for the 8% Pantene foams the values of $\ell/a_1 < 0.662$ in TABLE 6.5 show that all the plateaus can be characterized as quasi-steady state plateaus. The duration of these plateaus is much longer than those of the steady plateaus 1-3 in TABLE 6.2 for the 2% Pantene foam, and in TABLE 6.4 for the 4% Pantene foam.



Fig. 6.13. The evolution of the 8% Pantene thread with 3 mm thread length.

THE VALUES OF C₁ AND C₁ CORRESPONDING TO DIFFERENT QUASI-STEADY STATE PLATEAUS IN FIG. 6.13, AND THE MAXIMUM DIFFERENCE (%) WITH THE RIGHT-HAND SIDE OF THE EXPERIMENTAL THREAD. 8% PANTENE CONCENTRATION WITH 3 MM THREAD LENGTH

Plateau No.	ℓ / a_1	$C_1^{'}$	$C_1^{"}$	Max. diff. (%)	Duration of stable thread (s)
16	0.26	3.645	0.315	5.4	9.5
17	0.26	3.755	0.315	3.2	12
18	0.26	3.755	0.315	11.8	10.2



Fig. 6.14. Foam thread with the 8% Pantene concentration and 3 mm length corresponding to different plateaus from Fig. 6.13: (a) plateau No. 16, (b) plateau No. 17 and (c) plateau No. 18.

6.4.4. 8% Pantene concentration with 0.05 g of PEO

To further stabilize the foam, 0.05 g of PEO was added to the 8% Pantene foam (Shao et al., 1988). The presence of the high molecular weight PEO makes the stirring time longer to create uniform foam as shown in TABLE 6.6. The evolution of the minimum thread diameter in the case of the 8% Pantene concentration with PEO (Fig.

6.15) is similar to that of the 8% Pantene foam without PEO (Fig. 6.13). The comparison of the experimental profile corresponding to plateau in Fig. 6.15 with the theoretically predicted one is illustrated in Fig. 6.16 and TABLE 6.6. The latter shows that the maximum differences in this case are 5% for the 3rd trial, though the maximum differences are about 10-20% for the other cases as shown. In this case, as before, the theoretical solution corresponding to C₁ was the closest to the experimental data. It is emphasized that for the 8% Pantene with 0.05 g of PEO foams the values of $\ell/a_1 < 0.662$ in TABLE 6.6 show that all the plateaus can be characterized as the quasisteady state plateaus. It is also emphasized that the presence of PEO results in the longest-living plateaus 19-21 compared to those for the quasi-steady state plateaus 1-3 and 13-18 in TABLE 6.2, 6.4 and 6.5.



Fig. 6.15. The evolution of the 8% Pantene with 0.05 g of PEO thread with 3 mm thread length.

THE VALUES OF C_1 AND C_1 CORRESPONDING TO DIFFERENT QUASI-STEADY STATE PLATEAUS IN FIG. 6.15, AND THE MAXIMUM DIFFERENCE (%) WITH THE RIGHT-HAND SIDE OF THE EXPERIMENTAL THREAD. 8% PANTENE CONCENTRATION AND 0.05 G OF PEO WITH 3 MM THREAD LENGTH

Plateau No.	ℓ / a_1	Ċ	$C_1^{"}$	Max. diff. (%)	Duration of stable thread (s)
19	0.42	2.155	0.405	20.2	13.5
20	0.22	4.535	0.285	14.2	15.9
21	0.18	5.465	0.265	5.1	15.8



Fig. 6.16. Foam thread with the 8% Pantene concentration and 0.05 g of PEO and 3 mm length corresponding to different plateaus from Fig. 6.15: (a) plateau No. 19, (b) plateau No. 20 and (c) plateau No. 21.

6.5. Conclusion

The experiments show that relatively stable foam threads can be achieved with 8% Pantene concentration in water. The addition of 0.05 g of PEO does enhance thread stability even more. The increase in the Pantene concentration and the addition of PEO results in progressively longer-living quasi-steady foam threads. The theory was

developed to describe the steady-state shapes of foam threads. The theoretically predicted maximum length of the steady thread is in good agreement with the experimental observations. The theoretically predicted steady-state thread profiles agree with the experimental data with the maximum difference being in the range of 3-11%.

7. ELONGATIONAL & SHEAR RHEOLOGY OF SOAP FOAM

7.1. Introduction

Soap foam has a wide variety of industrial applications, one of them as a component in gypsum slurries, which helps make the resulting wallboards lighter, as in the technology adopted by the United States Gypsum Company (USG). There are two different types of soaps used by USG: unstable and stable. The main difference between the two types of soap is in the way that the molecules attach to each other. Unstable soap molecules attach to each other to create larger bubbles. The creation of large bubbles generates large sections of gypsum slurry between the bubbles. The large sections of gypsum slurry have experimentally been shown to withstand more stress, hence creating a stronger wallboard. Stable soap does not coalesce when turned into foam creating a fine core structure, while unstable soap creates a more stable structure when turned into foam.

The present work deals with both types of soap and foams made from them without addition of gypsum stucco. Therefore, not only soap is termed as stable or unstable in the present work but also the corresponding foams are called unstable or stable. Foam density is approximately 0.06 g/cm^3 and more than 90% of its volume represent itself as voids (bubbles) filled with air. The aim of this work is to establish and characterize the rheology of soap foam used at USG. The rheological tests were done using foams with different air content in stable foam and in combinations of stable and unstable foam with the same air content. The experimental data were processed using the power-law rheological constitutive equation. It was shown that this rheological constitutive equation

allows fitting the data obtained in the elongational experiments and finds the appropriate values of the consistency index K and the exponent n.

The experiments are described in section 7.2 and results are presented and discussed in section 7.3. Conclusions are drawn in section 7.4.

7.2. Experimental

7.2.1. Foam generation

Two different types of soap, stable and unstable, were used in this study to prepare foam. These soaps were mixed with water to make a 0.5% v/v soap solution. For the testing of foam resulting from stable soap, four different air contents were employed. For the rheological testing of foam resulting from a mixture of stable and unstable soap, three different mixing proportions between stable and unstable soap at the same air content were studied. All the combinations explored in the present work are listed in TABLE 7.1. The soap solutions were put in the foam generator provided by USG where air was added at different flow rates to generate different types of foam specified in TABLE 7.1. While the foam generator was working, the foam density was measured using a 300 mL beaker and a scale.

TABLE 7.1

SOAP SOLUTIONS CORRESPONDING TO DIFFERENT AIR FLOW RATES AND MIXING RATIOS

Case	Soap mixing Stable (%): Unstable (%)	Soap content (%v/v)	Soap solution Flow Rate (lb/hr)	Air flow rate (LPM)	Foam density (g/cm ³)
1	100:0	0.5	17.8	1.2	0.081
2	100 : 0	0.5	17.8	1.5	0.066
3	100 : 0	0.5	17.8	1.8	0.056
4	100 : 0	0.5	17.8	2.1	0.047
5	75:25	0.5	17.8	1.5	0.062
6	50 : 50	0.5	17.8	1.5	0.063
7	25:75	0.5	17.8	1.5	0.063

7.2.2. Elongational experiments with foam

The elongational rheometer, a CCD camera, and a continuous light source were assembled in the setup sketched in Fig. 7.1. A small transparent glass cup was filled with soap foam and placed on the lower part of the elongational rheometer as shown in Fig. 7.2. Then the cone-shaped tip of the rheometer was fully immersed into the foam in the cup. After that, the cone-shaped tip was released using the solenoid and moves up abruptly. After its motion had stopped, a foam filament was created and surface tension caused the elongational flow in the filament. As a result, the filament thinned. A high speed digital camera (Redlake MotionPro) recorded the filament self-thinning process with resolution of 512x1024 pixels. The video recording rates were 500 fps at a shutter speed of 1/3,000 s. At these rates only the last eight seconds can be recorded before filament break-up. An example is shown in Fig. 7.3 where the filament thinning corresponds to negative values of time and t=0 at the moment of filament breakup. To ensure statistical reliability of results, it was planned to perform 30 trials of the elongation tests for each case in TABLE 7.1. It appears that the first 4 cases were completed with improper illumination and had to be discarded. After solving the lighting problem, the mixtures of stable and unstable foams listed in TABLE 7.1 were studied in the elongational rheometer with 30 trials each. These trials had a very good consistency in the n-values (the exponent of the power law). It was decided that only 10 trials each would be sufficient for retesting of the first four cases. The recorded filament thinning images were transformed onto a PC. They were then processed in an automated mode with a MATLAB-based image processing program developed to obtain the evolution of the minimal filament diameter with time. The time required to process 4,000 separate images followed by data fittings was about 2 minutes on a 3.3-GHz Pentium-4 processor computer.



Fig. 7.1. Schematic of the elongational rheometer (Yarin et al., 2004; Tiwari et al., 2009).



Fig. 7.2. Detail of the elongational rheometer.



Fig. 7.3. The images of foam filament during its self-thinning (negative time values) up to the moment of breakup at t=0 s. (a) -8 s, (b) -6 s, (c) -4 s, (d) -2 s, (e) 0 s.

7.3. Results and Discussion

7.3.1. Results for the Elongational Flow

It can be seen that Fig. 7.4 shows that small steps are noticeable. We believe these steps result from bubbles popping on the thread surface followed by reorganization of remaining neighboring bubbles recreating a stable formation. In the other words, these small steps are related to a real physical effect.



Fig. 7.4. Diameter of a filament of 100% stable soap at air content of 1.8 LPM.

An example of the video images underlying the data summarized in TABLEs 7.2 and 7.3 is shown in Fig. 7.5 which corresponds to the data in Fig. 7.6.



Fig. 7.5. The video images of 100% stable soap foam at air content of 1.8 LPM. The self-thinning process corresponds to the negative values of time, while the moment of filament breakup is t=0 s. (a) -8 s, (b) -7 s, (c) -6 s, (d) -5 s, (e) -4 s, .(f) -3 s, (g) -2 s, (h) -1 s, (i) 0 s.

7.3.2. Processing results and rheological parameters

The experimental data in TABLEs 7.2 and 7.3 for different foams acquired at different foam and air compositions were processed using the Ostwald–de Waele power law (Astarita and Marrucci 1974)

$$\boldsymbol{\sigma} = -\mathbf{p}\mathbf{I} + \boldsymbol{\tau}, \quad \boldsymbol{\tau} = 2\mathbf{K} \left[2\mathrm{tr} \left(\mathbf{D}^2 \right) \right]^{(n-1)/2} \mathbf{D}$$
(7.1)

where σ is the stress tensor, τ is the stress deviator tensor, p is pressure, **D** is the rate-ofstrain tensor, **I** is unit tensor, K is the consistency index and n is the exponent (the flow behavior index).

TABLE 7.2

AVERAGE VALUES FOR STABLE FOAMS WITH DIFFERENT AIR CONTENT: ELONGATION TEST

Air flow rate (# of Trials)	2.1 LPM (9)	1.8 LPM (5)	1.5 LPM (5)	1.2 LPM (9)
Foam density (g/cm ³)	0.047	0.056	0.066	0.081
The exponent n	0.315	0.310	0.208	0.212
Standard deviation of n	0.045	0.021	0.006	0.041
Initial diameter (cm)	0.135	0.128	0.112	0.097
Consistency index K (CGS)	587	622	908	1030
Standard deviation of K	76	61	69	98

AVERAGE VALUES FOR DIFFERENT MIXTURES OF STABLE AND UNSTABLE

Percent of stable foam	100%	75%	50%	25%
(# of Trials)	(5)	(20)	(18)	(20)
Foam density (g/cm ³)	0.066	0.061	0.063	0.063
The exponent n	0.208	0.360	0.347	0.304
Standard deviation of n	0.006	0.067	0.053	0.036
Initial diameter (cm)	0.112	0.139	0.140	0.135
Consistency index K (CGS)	908	521	544	607
Standard deviation of K	69	103	98	78

FOAMS WITH AIR CONTENT OF 1.5 LPM: ELONGATION TEST

The experimental data in TABLEs 7.4 and 7.5 for different foams acquired at different foam and air content were found in shear experiments by measuring the shear viscosity for a given shear rate using the Brookfield shear viscometer. The viscosity and shear rate were used to find the K and n values using the shear projection of the power law model (7.1)

$$\mu_{\rm shear} = K \dot{\gamma}^{\rm n-1} \tag{7.2}$$

where μ is the shear viscosity, K is the consistency index, $\dot{\gamma}$ is the shear rate and n is the exponent (the flow behavior index).

Fitting the curves corresponding to Eq. (7.1) in the case of the uniaxial elongation (cf. Yarin et al., 2004) to the experimental data similar to those of Fig. 7.4, the values of K and n were found for foams with different compositions and air content. The surface tension used in these calculations is 60.7 mN/m. This was measured using USG's ring-type tensiometer using 100% stable soap with 1.5 LPM air content. The results obtained are discussed below. In TABLEs 7.2-7.5 the number of trials averaged to generate the parameter values is listed in parentheses.

TABLE 7.4

AVERAGE VALUES FOR STABLE FOAMS WITH DIFFERENT AIR CONTENT: SHEAR TEST

Air flow rate	2.1 LPM	1.8 LPM	1.5 LPM	1.2 LPM
(# of Trials)	(7)	(7)	(5)	(6)
Foam density (g/cm ³)	0.047	0.056	0.066	0.081
The exponent n	0.73	0.63	0.34	0.57
Standard deviation of n	0.04	0.08	0.06	0.09
Consistency index K (CGS)	2.0	2.8	6.4	3.1
Standard deviation of K	0.3	0.6	1.1	1.0

TABLE 7.5

AVERAGE VALUES FOR DIFFERENT MIXUTRES OF STABLE AND UNSTABLE

Percent of stable foam	100%	75%	50%	25%
(# of Trials)	(5)	(8)	(6)	(7)
Foam density (g/cm ³)	0.066	0.061	0.063	0.063
The exponent n	0.34	0.74	0.32	0.24
Standard deviation of n	0.06	0.02	0.16	0.09
Consistency index K (CGS)	6.4	2.1	4.7	7.5
Standard deviation of K	1.1	0.1	2.8	1.4

FOAMS WITH AIR CONTENT OF 1.5 LPM: SHEAR TEST







Fig. 7.6. The viscosity calculated based on the rheological parameters established using the results. (a) The elongational rheometer with different air content in stable soap foam. (b) The elongational rheometer with different proportion of stable and unstable soap foam. (c) The shear viscometer with different air content in stable soap foam. (d) The shear viscometer with different proportion of stable and unstable soap foam.

As expected, the foam density increases as the air content is reduced. Foam density stays constant regardless of the proportion of stable to unstable foam showing that stable and unstable foam have equal densities. With stable soap foam, the n-value decreases with the reduction of air content. For foam consisting of mixed soap, the n-value decreases with the increase of unstable soap. Before the unstable soap is introduced, the average n-value is 0.208 (TABLE 7.3) and after creating a mixture with only 25% unstable foam the n-value rises to 0.36. The K-value increases with a decrease in air content in stable soap foam. Introducing unstable soap into the mixture reduces the K value but the amount of unstable soap does not affect the K-value significantly. The data reflects approximately the last 8 seconds before filament breakup. The initial diameter is measured at the beginning of this time frame. The initial diameter is attributed to the diameter 8 seconds before breakup. More air content in the soap foam causes the initial diameter to be larger. Because the time of breakup for all cases is approximately the same, the rate of thinning is faster for the filament with the larger initial diameter. In mixtures of stable and unstable soap the amount of air content is held constant causing the initial diameter to be about the same. This shows that the rate of breakup is the same for all mixtures of stable and unstable soap with the same air content. The averaged values, such as n, the initial diameter, and K, of stable soap foam with air content 2.1 LPM are similar to those of the mixture of stable and unstable soap foam with air content of 1.5 LPM.

There is a large disparity between the values of K found from the elongation tests as compared to those found in shear tests. This probably stems from jamming of the thin foam filament by bubbles; the topic is still under investigation. The values of n found are also different in the elongation and shear tests.

7.4. Conclusion

Soap characteristics of different mixtures of steady and unsteady soaps and varying air contents were studied. It was found that the soap density and the exponent n values in the power-law rheological constitutive equation both decrease with the reduction of air content in stable soap foam only. In mixed soap foam, a small introduction of unstable soap causes a large increase in the n value and a large decrease in the K value. However, further addition of unstable soap causes the n value to decrease while the K value increases. A further investigation of the reasons of too high effective viscosities found for foams is needed.

8. POOL BOILING ON NANO-TEXTURED SURFACES

8.1. Introduction

Pool boiling is known as a very effective mechanism of two-phase heat transfer due to its high heat flux values. The regimes of pool boiling can be divided on the boiling curve as free convection boiling, nucleate boiling, transition boiling, and film boiling (Nukiyama, 1966). Among them nucleate boiling is considered to be the most desirable heat transfer regime, since it is associated with high heat fluxes at low superheat surface temperature, which is attractive for such practical applications as cooling of electronics, boilers, nuclear and chemical reactors, and refrigeration systems. However, there exists a critical value of heat flux called critical heat flux (CHF) at which is nucleate boiling transforms into film boiling. The latter involves heat transfer through coolant vapor rather than through liquid coolant itself, which is a heat transfer mechanism. There were multiple efforts to increase CHF and boiling heat transfer coefficient (BHT), which is by definition the heat flux divided by the corresponding superheat temperature.

The aim of the present work is in the experimental evaluation of the effect of nanotextured electrospun coatings on the enhancement of BHT coefficient and CHF in pool boiling of water and ethanol. The pool boiling experiments are described in section 8.2, and the corresponding theory is developed in section 8.3. The results are presented and discussed in section 8.4. Conclusions are drawn in section 8.5.

8.2. Experimental

8.2.1. Materials

Polyacylonitrile (PAN, $M_w = 150$ kDa) was obtained from Polymer Inc., N-Dimethylformamide (DMF) anhydrous 99.8%, sulfuric acid, hydrochloric acid, copper sulfate, and formaldehyde were obtained from Sigma-Aldrich. A copper rod obtained from McMaster-Carr; its surface was polished using Norton 500 Grit T421 sandpaper (average particle diameter; 20 µm).

8.2.2. Electrospinning of PAN solution

PAN was mixed with DMF to prepare 12 wt% PAN solution in DMF. The solution was put on a hotplate and magnetically stirred for a few hours to make the solution homogeneous. A standard electrospinning setup (Reneker et al., 2000; Reneker et al., 2007; Reneker et al., 2008) was used to prepare PAN nanofiber mats. Fibers were electrospun for 1 min onto the top surface of an edge of a copper rod (25 mm in dia). During electrospinning the solution flow of 0.7 ml/hr was sustained and the process was conducted at the electric field strength of 1.1 kV/cm.

8.2.3. Copper-plating of PAN nanofiber mats

The electric conductivity of the polymer nanofiber mats is insufficient to use them as an electrode in the electroplating process. To enhance the electric conductivity, the nanofiber mats were sputter-coated with Pd to a thickness of 15 nm by using a 120 Cressington sputter controller. A few drops of electroplating solution were dripped onto the sputter-coated nanofibers to pre-wet them and facilitate electroplating. Then the sputter-coated nanofiber mats were immersed in the copper-plating solution which consisted of sulfuric acid (5 g), hydrochloric acid (0.5 g), copper sulfate (16 g), formaldehyde (10 g), and DI water (100 ml). The Pd-coated PAN nanofibers were used as a cathode immersed into the electroplating bath. A bare copper substrate served as an anode. A laboratory electroplating station HSEPS-10 with a cathode stand was used to electroplate nanofiber mats. The electric current density was kept at 100 mA/cm² for 2 min to form a copper coating on the fibers without blocking the inter-fiber pores. Then the copper-plated nanofiber mats were extracted from the electroplating bath and put into 10% formaldehyde solution for 5 min, and after that washed with DI water and dried.

8. 2.4. Optical Observations

All the observations were done using optical microscope (Olympus BX-51) and scanning electron microscope (JEOL JSM-6320F). Prior to observations by scanning electron microscopy, all the samples were sputter coated to a thickness of 7-8 nm. The surface topography of the samples was observed by SEM and optical microscope (Fig. 8.1), where Figs. 8.1a and 8.1b show the surface morphology of the bare copper surface and Figs. 8.1c and 8.1d depict the surface morphology of the nanofiber-coated samples. In case of bare copper the roughness results from the inevitable scratches of the surface. On the other hand, for nanofiber-coated surfaces roughness is associated with the interfiber pores, and on a smaller scale with the secondary structures on the individual copper-plated nanofiber surface as was shown in Sinha-Ray et al. (2011) and is seen in Fig. 8.1. Figs. 8.1a and 8.1b show that the maximum depth of scratches on the bare copper surface was about 3 µm and the length between two neighboring grooves was about 6-40 µm.

The nanofiber mats used in the present work were about 100 μ m thick and the inter-fiber pore diameter was about 3-10 μ m. The pitch of the secondary structures on the surface of an individual metal-plated nanofiber was about 20-100 nm as seen in Figs. 8.1c and 8.1d.



Fig. 8.1. Microstructure images of bare copper and copper-plated nanofiber-covered surfaces. (a) SEM image of bare copper surface. (b) Optical microscope image of bare copper surface. (c) SEM images of copper-plated nanofiber-covered surface. (d) SEM image of an individual copper-plated nanofiber from the encircled area in panel (c).

Overall, the nanofiber-covered surfaces were significantly rougher than those of the bare copper samples. In fact, the nanofiber-covered surfaces were fluffy.

The static contact angles on bare copper and copper-plated nanofiber-covered surfaces were measured at room temperature for both water and ethanol. To minimize the gravity effects, droplets were softly deposited from G30 needle (ID=0.159 mm) to avoid any dynamic effects discussed in Sinha-Ray et al. (2011). The value of the static contact angles of water on bare copper surface was 78° , whereas it was 108° on the copper-plated nanofiber-covered surface as shown in Fig. 8.2. The contact angles of ethanol were quite small for both bare copper and nanofiber-covered surfaces: 16° and 5° , respectively.



Fig. 8.2. Contact angles of water on (a) bare copper surface, and on (b) copper-plated nanofiber-covered surface.

8.2.5. Pool boiling experiment

In the experiments with pool boiling, two different surfaces were used as heating elements for comparison. The first one was a plain bare copper surface, whereas the second one was the copper surface with a 100 µm-thick copper-plated nanofiber mat on top of it (cf. Figs. 8.3-8.4). Two different setups were developed for these experiments. The first setup is depicted in Fig. 8.3. This experimental setup incorporated a ceramic hotplate (Cimarec, Max. temp. 540 °C), a Teflon ring surrounding the copper rod and a silica fabric sheet as an insulator underneath the ring, as well as three thermocouples T_1 , T_2 , and T_{fl} (Omega Inc., T type) which measured temperatures at a lower and upper points of the copper rod axis, and in the liquid bulk, respectively. The hotplate nominal temperature had been increased from 130 °C to 540 °C with a 10 °C increment at every 5-10 min. It is emphasized that the hotplate nominal temperature was kept for 30 min at 130 °C after the beginning of boiling to degasify the liquid. After each stepwise increase in temperature, the thermal field was able to reach steady state well before the end of a 5-10 min interval. The temperatures were recorded by a dual input HH86AW microprocessor thermometer (Omega Inc.) just before each new temperature increment, which means the measurements always corresponded to steady states over the whole temperature range studied. After measuring the temperatures, 5-15 ml of boiling liquid was added using a syringe filled from a liquid reservoir kept at another hotplate to sustain a constant liquid level during the entire experiment, since in the first setup liquid vapor was released to the atmosphere. The liquid volume in the first setup was 25 ml.



Fig. 8.3. Sketch of the pool boiling experimental setup (the first setup).

The temperature field in the copper rod was assumed to be one-dimensional, since heat loss through the Teflon ring was very small (the thermal conductivity of Teflon is only 1/1743 of that of copper). The heat flux was calculated by means of the Fourier's Law

$$q'' = -k \frac{dT}{dx}$$
(8.1)

where x is the coordinate reckoned along the rod axis, and q" is the heat flux and k is the thermal conductivity of copper; $k=401 \text{ W/(m \cdot K)}$. The derivative dT/dx was evaluated

using the measured temperature values as $\Delta T/\Delta x$, with $\Delta T=T_1 - T_2$ being measured by the thermocouples T_1 and T_2 , and the axial inter-thermocouple distance Δx being equal to 8.5 mm. The superheat, $\Delta T_{sat} = T_w - T_{sat}$ is computed as

$$\Delta T_{\text{sat}} = T_2 - \frac{(T_1 - T_2)\Delta x_w}{\Delta x} - T_{\text{fl}}$$
(8.2)

where $\Delta x_w = 4.8$ mm is the distance between the locations with temperatures T_2 and T_w . In Eq. (8.2) $T_w = T_2 - (T_1 - T_2)\Delta x_w / \Delta x$ and $T_{sat} = T_{fl}$.

The second setup was developed to eliminate the need for liquid replenishment. This setup incorporated a vapor condensing unit (Fig. 8.4). The condensate was returned to the liquid bulk, which allowed us to keep liquid level constant. A copper plate was added on top of the ceramic hotplate to increase the heat flux. The experimental procedure in the second setup was identical to the one in the first setup, except liquid replenishment steps were eliminated. It is emphasized that the liquid volume in the second setup was higher than in the first one, namely 250 ml. An immersion heater (Hotwatt, 200W) could be inserted into the second setup as an auxiliary heat source to facilitate boiling as shown in Fig. 8.4.



Fig. 8.4. Sketch of the pool boiling experimental setup (the second setup). In some experiments an immersion heater was inserted as shown to facilitate boiling.

8.3. Theoretical Model

8.3.1. Nucleation rate and bubbling frequency

In the process of nucleate boiling, heterogeneous nucleation takes place at the solid-liquid interface. For heterogeneous nucleation Forster and Greif postulated that bubbles act as microscopic pumps, which draw cold ambient fluid to the surface as the

bubble releases or collapses. They further assumed that each bubble pumps a quantity of liquid equal to its volume at release. The mean heat flux from the surface is then given by

$$q'' = \rho_{l} c_{pl} \left(\frac{2\pi}{3}\right) R_{max}^{3} \left(\frac{1}{2}\right) (T_{w} - T_{sat}) fn_{a}^{'}$$
(8.3)

where ρ_1 is the liquid density, c_{pl} is the liquid specific heat, R_{max} is the maximum bubble radius at release, T_w is the wall temperature, T_{sat} is the saturation temperature, f is the bubble release/departure frequency, and n'_a is the density of active nucleation sites at the surface.

Cole and Rohsenow proposed the following correlation for the bubble departure diameter which yields a good agreement in the entire range of experimental conditions for a variety of fluids

$$d_{d} = 2R_{max} = A_{1} \left[\frac{\sigma}{g(\rho_{1} - \rho_{v})} \right]^{1/2} \left(\frac{\rho_{1}c_{pl}T_{sat}}{\rho_{v}L} \right)^{5/4}$$
(8.4)

where the dimensionless factor $A_1 = 1.5 \times 10^{-4}$ for water and 4.65 x 10^{-4} for the other fluids, σ is the surface tension, g is gravity acceleration, ρ_v is vapor density, and L is latent heat of evaporation.

Mikic and Rohsenow (1969) used the growth of a thermally controlled bubble in a non-uniform temperature field near a heated surface to evaluate the waiting and bubble growth times and proposed the following relation for the bubble departure frequency

$$f^{1/2}d_{d} = \left(\frac{4}{\pi}\right)Ja\sqrt{3\pi\alpha_{1}}\left[\left(\frac{t_{g}}{t_{d}+t_{g}}\right)^{1/2} + \left(1+\frac{t_{g}}{t_{d}+t_{g}}\right)^{1/2} - 1\right]$$
(8.5)

where Ja is the Jacob number, α_1 is the thermal diffusivity of fluid, t_g is the bubble growth time, t_d is the delay time.

They further showed that in the range $0.2 < t_d / (t_d + t_g) < 0.8$, the above equation is approximated by the following simpler form

$$f^{1/2}d_d = 0.83 Ja \sqrt{\pi \alpha_1}$$
 (8.6)

In Eqs. (8.5) and (8.6) the Jacob number Ja is defined by

$$Ja = \frac{(T_{w} - T_{sat})c_{pl}\rho_{l}}{\rho_{v}L}$$
(8.7)

The density of nucleation sites at the surface is given by Mikic and Rohsenow

$$\mathbf{n}_{a} = \mathbf{A}_{2} \left[\mathbf{R}_{0} \frac{\boldsymbol{\rho}_{v} \mathbf{L}}{2\sigma \mathbf{T}_{\text{sat}}} \right]^{m} \left[\mathbf{T}_{w} - \mathbf{T}_{\text{sat}} \right]^{m}$$
(8.8)

where R_0 is the threshold radius for which n_a would be one per unit surface area. In Eq. (8.8) A_2 is a dimensional constant found below.

Combining Eqs. (8.3) (8.4), (8.6) and (8.8) yields the following expression for the heat flux

$$q'' = B(T_w - T_{sat})^{3+m}$$
 (8.9)

where

$$\mathbf{B} = 0.83^{2} \left(\frac{\pi^{2}}{12}\right) \alpha_{1} A_{1} A_{2} \left[\frac{\sigma^{(1/2-m)}}{\sqrt{g(\rho_{1}-\rho_{v})}}\right] \left(\rho_{1} c_{p1}\right)^{17/4} \left[\frac{T_{sat}}{\rho_{v} L}\right]^{5/4-m} \left(\frac{R_{o}}{2}\right)^{m}$$
(8.10)

Equations (8.9) and (8.10) are similar to Eq. (8.12) in Mikic and Rohsenow (1969), albeit in the latter instead of Eq. (8.5) a different expression was used, which made their final result different from the present one.

Forster and Greif assumed that the mean temperature of the warm liquid pumped away from the surface is equal to the average of the wall and bulk temperature. Based on that assumption T_w for bare copper is $T_{w,b} = (T_w + T_{sat})/2$. For nanofiber mats the surface temperature T_w can be taken as $T_{w,fi} = \beta T_w + (1-\beta)T_{sat}$ where β is a dimensionless overheat factor. If $1/2 < \beta \le 1$, $T_{w,fi} > T_{w,b}$ which will correspond to the intensification of boiling at the nano-textured surfaces compared to the corresponding bare surfaces. This, indeed, can happen since bubbles grow inside the nanofiber mats with metalized fibers having the same temperature as the base; cf. Fig. 8.5.



Fig. 8.5. A vapor bubble formed at a liquid-solid interface. (a) Bare surface, (b) surface covered by nanofiber mat.

Therefore, using Eq. (8.9), one can express the heat flux at the bare surface as

$$q_{b}^{"} = B \left[\frac{1}{2} (T_{w} - T_{sat}) \right]^{3+m}$$
 (8.11)

and the heat flux in the case of nanofiber mats as

$$\mathbf{q}_{\mathrm{fi}}^{"} = \mathbf{B} \left[\beta (\mathbf{T}_{\mathrm{w}} - \mathbf{T}_{\mathrm{sat}}) \right]^{3+m}$$
(8.12)

Form Eqs. (8.11) and (8.12), the ratio of the heat fluxe of a surface covered by nanofiber mat to that of a bare surface becomes

$$q_{\rm fi}^{"}/q_{\rm b}^{"} = (2\beta)^{3+{\rm m}}$$
(8.13)

The corresponding heat transfer coefficients h are defined as

$$h_{b} = \frac{q_{b}^{'}}{T_{w} - T_{sat}} = \frac{1}{2} B \left[\frac{1}{2} (T_{w} - T_{sat}) \right]^{2+m}$$
(8.14)

$$h_{fi} = \frac{q_{fi}}{T_w - T_{sat}} = \beta B \left[\beta (T_w - T_{sat}) \right]^{2+m}$$
(8.15)

where h_{b} , and h_{fi} are the heat transfer coefficients of the bare surface and a surface covered with nanofiber mat, respectively.

From the above equations, the ratio of the heat transfer coefficient on a bare surface to that on a surface covered with nanofiber mat reads

$$h_{fi}/h_{b} = (2\beta)^{3+m}$$
 (8.16)

The values of β can be evaluated theoretically, as is described below in the following subsection, where the theoretically predicted values of β are in the range 0.834-0.972.

8.3.2. Liquid temperature inside fluffy nanofiber mats

Consider the temperature field in liquid in contact with a hot wall assuming that it is in steady state and governed by the planar Laplace equation. We assume that the wall is
isothermal and had periodic protrusions into liquid in the form of star shapes representing nanofiber mats of different degree of fluffiness n, as shown in Fig. 8.6.



Fig. 8.6. Nanofiber mat morphologies of different degree of fluffiness n.

A generic star-like shape mimicking nanofiber mat fluffiness is shown in Fig. 8.7a. The dotted liquid domain in the physical plane of the complex variable z=x+iy (with x and y being the Cartesian coordinates and i the imaginary unit) surrounding the star-like structure can be conformally mapped onto the dotted upper half-plane in the ζ - plane (Fig. 8.7b; $\zeta=\xi+i\eta$) by the following mapping function

$$z = \frac{1}{\sqrt[n]{4}} \frac{\left\{ \left[\left(i - \zeta \right) / \left(i + \zeta \right) \right]^n + 1 \right\}^{2/n}}{(i - \zeta) / (i + \zeta)}$$
(8.17)

where the degree of fluffiness n is a positive integer.



Fig. 8.7. (a) Conformal mapping of the area in the physical plane z surrounding the starlike fluffy structure onto (b) the upper half-plane ζ .

In the ζ -plane the temperature distribution is one-dimensional

$$T(\xi,\eta) = T_{w} - \frac{qL\eta}{k}$$
(8.18)

where T_w is the wall temperature, k is the thermal conductivity of liquid, L is the length scale, and q is the heat flux.

The temperature field (Fig. 8.7) satisfies the planar Laplace equation. Therefore, we can introduce the holomorphic complex thermal potential

$$\chi = \psi(\xi, \eta) + iT(\xi, \eta) \tag{8.19}$$

with the real part ψ being related to the imaginary part (temperature) T via the Cauchy-Riemann conditions

$$\frac{\partial \Psi}{\partial \xi} = \frac{\partial \Gamma}{\partial \eta} \tag{8.20}$$

$$\frac{\partial \Psi}{\partial \eta} = -\frac{\partial T}{\partial \xi}$$
(8.21)

Combining Eqs. (8.18) and (8.19), we find the complex thermal potential in the ζ - plane as

$$\chi(\zeta) = iT_{w} - \frac{qL}{k}\zeta$$
(8.22)

On the other hand, Eqs. (8.22) and (8.17) allow one to find the complex thermal potential in the z-plane $\chi(z)$ and its imaginary part T(x,y), which represents temperature field about the star-like fluffy structures of Fig. 8.6, when their rays are sustained at the wall temperature T_w and the heat flux at infinity is q. The temperature fields produced by Eqs. (8.22) and (8.17) are illustrated in Fig. 8.8.















Fig. 8.8. The temperature fields affecting the growing bubbles versus the number of rays (the degree of fluffiness): (a) n=3, (b) n=4, (c) n=5, (d) n=6, (e) n=7, (f) n=8, (g) n=9, (h) n=10, (i) n=11, (j) n=12. The length scale is taken as the ray length L.

The average temperature affecting the growing is calculated as

$$T_{av} = \frac{\iint\limits_{S} T dx dy}{S}$$
(8.23)

where the integral is evaluated over the inter-ray areas in Fig. 8.8.

The average temperature values for different degrees of fluffiness n are presented in TABLE 8.1.

TABLE 8.1

DIMENSIONLESS AVERAGE TEMPERATURE FOR DIFFERENT VALUES OF THE

DEGREE OF FLUFFINESS n

Degree of fluffiness	n=3	n=4	n=5	n=6	n=7	n=8	n=9	n=10	n=11	n=12
Average temperature	0.834	0.889	0.922	0.939	0.948	0.955	0.961	0.966	0.970	0.972

8.4. Results and Discussion

In pool boiling experiments the wall superheat temperature (ΔT_{sat}) is the temperature difference between the top surface of the copper rod (the wall temperature) and liquid saturation temperature [cf. Eq.(8.2)]. The wall temperature was obtained by linear extrapolation of two measured temperatures T₁ and T₂ measured as shown in Figs.

8.3 and 8.4. The experimental heat flux q" was determined using Eq. (8.1). The measured heat flux values were plotted versus the wall superheat temperature in both cases of bare copper surface and copper surface coated with copper-plated nanofiber mat. Ethanol and water were used as boiling liquids.

8.4.1. Delamination of nanofiber mats

When nanofiber mats were too thick, it was found that they were insufficiently bonded to the copper substrate during electroplating. That created problems in the experiments with boiling. In particular, when the substrate temperature had been increased and boiling became more severe, some parts of the nanofiber mats delaminated from the substrate, as is seen in Fig. 8.9. The effect of the nanofiber delamination on the measured boiling curve is illustrated in Fig. 8.10.



Fig. 8.9. Three copper substrates with delamination of copper nanofiber mats seen on top of them.



Fig. 8.10. Experimental data from the first setup for pool boiling of ethanol over the smooth bare copper surface and the same surface covered with copper-plated nanofiber mat. In one of the two cases shown nanofiber mat delaminated from the substrate. The effect of the delamination was in a significant reduction of the heat flux removed from the surface and a significant increase in the surface temperature.

8.4.2. Results for ethanol

For the error analysis and to achieve reliable results, several trials were done for each bare copper surface and metalized nanofiber-covered surface as shown, for example, in Fig.8.11. It was found that the nanofiber-covered samples differ significantly, which is determined by the intrinsic variability of the nanofiber mats. Therefore, the results obtained with different nanofiber-coated samples revealed a larger variability than those with the bare copper samples. Fig. 8.11 illustrates the sample-to-sample variance, which would be the dominant source of the overall variance in the measured values. It is clearly seen that in any case the results corresponding to the nanofiber-coated surfaces are qualitatively similar to each other and quite distinct from those corresponding to the bare copper surfaces.



Fig. 8.11. Pool boiling data for three different samples of metalized nanofiber-covered surfaces and two bare copper surfaces from the second setup for ethanol. The results illustrate the effect of the sample-to-sample variation.

The experimental results show that the heat flux and heat transfer coefficient are much higher at the same superheat temperature on nanofiber-covered surface than on the corresponding bare surface, as is seen in Figs. 8.12-8.15. This means that the heat removal is much higher due to the presence of the nanofiber mats. This effect is due to the fact that the nanofiber-covered surfaces promote bubble growth by increasing the average temperature of liquid surrounding growing bubbles (cf. subsection 8.3.2). The experimental data were fitted to Eqs. (8.11) and (8.12). The value of m=1/2 was found for the data produced by both setups for both boiling on bare and nano-textured surfaces as listed in TABLE 8.2. This means that the heat flux $q'' \sim (\Delta T_{sat})^{3.5}$, which is similar to Mikic and Rohsenow (1969). By fitting Eq. (8.12) to the data for boiling on nanotextured surfaces in Figs. 8.12 and 8.13, the value of β was found in the range 0.85-0.92. On the other hand, the theoretical results in subsection 8.3.2 reveal the value of β = 0.889-0.922 corresponding to the degree of fluffiness n=4-5 (defined in subsection 8.3.2). Therefore, in the case of ethanol the experimental values for β are in good agreement with the theoretical predictions for n=4-5. Correspondingly, according to Eq. (8.13) the heat flux with nanofiber mat was about 6-8 times larger than that of the corresponding bare surface at the same superheat temperature.



Fig. 8.12. Pool boiling data from the first setup for ethanol. The corresponding value of $\beta = 0.92$. CHF has not been reached in the experiment with ethanol in the first setup because of the limited heat flux available. The maximum heat flux available was reached in the present experiment.



Fig. 8.13. The heat transfer coefficient measured using the first setup for ethanol on a smooth bare copper surface and nanofiber mat.

TABLE 8.2

PARAMETERS IN EQS. (8.11) – (8.16) FOR THE HEAT FLUX AND HEAT TRANSFER COEFFICIENT CORRESPONDING TO ETHANOL BOILING

Parameter	First setup	Second setup
m	0.5	0.5
β	0.92	0.85
$q_{ m fi}/q_{ m b}$	8.45	6.41
h _{fi} /h _b	8.45	6.41



Fig. 8.14. Pool boiling data from the second setup for ethanol on a smooth bare copper surface and nanofiber mat. The maximum heat flux was reached in the present experiment. Namely, CHF has been reached with second setup.



Fig. 8.15. The heat transfer coefficient measured using the second setup with ethanol on a smooth bare copper surface and nanofiber mat.

8.4.3. Results for water

The temperature of boiling water was taken as 100 °C in all cases. The experimental data for water discussed in the present subsection reveal that the heat flux and heat transfer coefficient on nanofiber mat are about 3-5 times larger than those on bare copper surfaces. Once again, the superiority of the metalized nanofiber-covered surfaces in comparison to the corresponding bare copper surfaces is due to the fact that the effective liquid temperature in the pores of the fluffy nanofiber-mat surfaces is higher than near the smooth ones. The value of the exponent m in Eqs. (8.9)-(8.16) was found to be

m=0.3~1.0 as listed in TABLE 8.3. That means that in the case of water (in distinction from the case of ethanol) the heat flux q" ~ $(\Delta T_{sat})^{3.3 \text{ to } 4}$ for boiling in both first and second setups. These exponent values are similar to those reported in Mikic and Rohsenow (1969). The experimental results for water on smooth copper surface and copper-plate nanofiber mats are presented in Figs. 8.16-19. By fitting Eq. (8.12) to the data for boiling on nano-textured surfaces in Figs. 8.16 and 8.18, the value of β was found in the range 0.66-0.76. On the other hand, the theoretical results in subsection 8.3.2 reveal the lowest value of β = 0.834 corresponding to the lowest degree of fluffiness n=3 of the nanofiber-covered surface. The difference in the experimental and theoretical ranges for β in the present case of boiling water is larger than that in subsection 8.4.2 for ethanol. This could be associated with the fact that for lower surface fluffiness of nanofiber mats in the present case, the bubble diameter becomes larger than the entire thickness of the nanofiber layers which do not cover the entire bubble in water.



Fig. 8.16. Pool boiling data from the first setup for water. CHF has not been reached in the experiment with water in the first setup because of the limited heat flux available. The maximum heat flux available was reached in the present experiment.



Fig. 8.17. The heat transfer coefficient measured using the first setup for water on a smooth bare copper surface and nanofiber mat.

TABLE 8.3

PARAMETERS IN EQS. (8.11) - (8.16) FOR THE HEAT FLUX AND HEAT TRANSFER COEFFICIENT CORRESPONDING TO WATER BOILING

Parameter	First setup	Second setup
m	1.0	0.3
β	0.76	0.66
$q_{ m fi}/q_{ m b}$	5.34	3.04
h _{fi} /h _b	5.34	3.04



Fig. 8.18. Pool boiling data from the second setup for water on a smooth bare copper surface and nanofiber mat. CHF could not be reached, since the heat supply was insufficient.



Fig. 8.19. The heat transfer coefficient measured using the second setup with water on a smooth bare copper surface and nanofiber mat.

The images of nucleate boiling on the bare copper surface and nanofiber-covered surface at the hotplate temperatures of 200 $^{\circ}$ C and 400 $^{\circ}$ C are shown in Fig. 8.20. The upper images (a,b) show isolated bubbles at the heat flux around 10 W/cm² and the lower images (c,d) show jets and columns at the heat flux around 40 W/cm².



Bare copper

Nanofiber mats



Bare copper

Nanofiber mats

Fig. 8.20. The images of pool boiling of water on the bare surfaces and surfaces coated with nanofiber mats at the hotplate temperature of 200 °C in panels (a,b) and 400 °C in panels (c,d).

8.4.4. Critical heat flux

The theoretical expression for CHF proposed in (Zuber, 1959; Lienhard et al., 1973) implies that the critical heat flux is attained when large bubbles are formed as the interfaces of large vapor columns leaving the surface become unstable due to the Kelvin-Helmholtz instability, whereas the vapor layer near the heater is subjected to the

$$\lambda_{\rm H} = \frac{3\pi\sigma}{\rho_{\rm v} V^2} \tag{8.24}$$

where σ is the surface tension, and V is the vapor velocity in the column.

The fastest growing wavelength of the Rayleigh-Taylor instability is obtained as (Chandrasekhar, 1961)

$$\lambda_{\rm D} = 2\pi \left(\frac{3\sigma}{\rho_{\rm I}\,g}\right)^{1/2} \tag{8.25}$$

By equality $\lambda_{\rm H} = \lambda_{\rm D}$ yields using Eqs.(8.24) and (8.25), the vapor velocity as

$$\mathbf{V} = \left(\frac{3\sigma\rho_1 g}{4{\rho_v}^2}\right)^{1/4} \tag{8.26}$$

Zuber's CHF model (Zuber, 1959), as refined by Lienhard and Dhir assumed that the cross-sectional radius R of the vapor column is equal to $\lambda_D/4$ and the centerline spacing of the column coincides with the nodes of the fastest growing wavelength of the Rayleigh-Taylor instability, λ_D (cf. Fig. 8.21).



Fig. 8.21. Vapor column spacing during boiling over a hot horizontal surface corresponding to Zuber's CHF model (Zuber, 1959; Carey, 2008).

For liquids at saturation temperature the thermal balance equation corresponding to heat removal from the hot surface by vapor columns breaking into big bubbles under the CHF conditions reads

$$q''_{max} S_{surface} = \rho_v LVS_{column}$$
(8.27)

where $q_{max}^{"}$ is the critical heat flux, $S_{surface} = \lambda_D^{2}$ is the unit cell surface area, ρ_v is the vapor density, L is the latent heat of vaporization, V is the vapor velocity, and $S_{column} = \pi \lambda_D^{2}/4$ is the vapor column cross-sectional area.

Therefore, the critical heat flux in pool boiling is found using Eqs. (8.25) and (8.27) as

$$q''_{max} = 0.182 \rho_v L \left(\frac{\sigma \rho_1 g}{\rho_v^2}\right)^{1/4}$$
 (8.28)

In the above analysis, in distinction from the original Zuber's model (Zuber, 1959; Lienhard et al., 1973; Carey, 2008) the exact expressions for the wavelengths of the fastest growing modes of the Kelvin-Helmholtz and Rayleigh-Taylor were used, and $\rho_1 - \rho_v$ is approximated as ρ_1 . Therefore, Eq. (8.28) differs from the original Zuber's expression

$$q''_{max} = 0.131 \rho_{v} L \left[\frac{\sigma(\rho_{1} - \rho_{v})g}{\rho_{v}^{2}} \right]^{1/4}$$
(8.29)

The critical heat flux (CHF) in ethanol boiling was reached in the second setup for both bare copper surface and nanofiber mat as shown in Fig. 8.22. It is seen that the CHF in boiling on both bare copper surface and nanofiber mat is close. However, the wall superheat is dramatically different namely, the wall temperature is about 10 °C lower on the nano-textured surface. The comparison of the experimental data with Eqs. (8.28) and (8.29) is presented in TABLE 8.4, which shows that the former agrees with the data better than the latter.



Fig. 8.22. Nucleate boiling data for ethanol on a smooth bare copper surface and nanofiber mat obtained using the second setup. The horizontal lines correspond to the CHF level.

TABLE 8.4

CHF FOR ETHANOL MEASURED IN THE SECOND SETUP FOR NUCLEATE BOILING ON BARE SURFACE AND NANOFIBER-COVERED SURFACE IN COMPARISON WITH THEORETICAL PREDICTIONS

Parameter	Eq. (8.28)	Eq. (8.29)	Bare copper	Nanofiber mat
CHF (W/cm ²)	60.9	43.7	60.1	54.5

Beyond CHF, as usual, the heat flux decreases as the wall superheat temperature increases which corresponds to the transitional boiling, in which the formation of vapor film reduces the heat flux. In the transition region the heat flux on nanofiber mats reduces much slower than on the corresponding bare copper surface, as shown in Fig. 8.23. That is because the bubble departure frequency and the density of nucleation sites on bare copper surface are much less than those on nanofiber mat. This implies that nanofiber mat tends to prevent formation of the vapor film.



Fig. 8.23. Nucleate boiling and the transition boiling data for ethanol on smooth bare copper surface and nanofiber mat in the second setup.

8.4.5. The effect of the immersion heater on water boiling in the second setup

In an effort to increase the maximum heat flux, an additional immersion heater was used in several experiments in the second setup. It turned out that its effect on the heat flux was insignificant for both bare surfaces and nanofiber-covered surfaces (cf. Fig. 8.24). The results showed only slightly higher heat fluxes achieved with the immersion heater compared to those without this heater.



Fig. 8.24. Nucleate boiling data for on smooth bare copper surface and nanofiber mat in the second setup with/without an additional immersion heater.

8.5. Conclusion

Nano-textured copper surfaces coated with copper nanofiber mats possess a 6-8 times higher heat flux and heat transfer coefficient in nucleate boiling of ethanol in comparison with those of the corresponding bare copper surfaces. For the nucleate boiling of water, the heat flux and heat transfer coefficient on nano-textured surfaces is 3-5 times higher than those of bare copper surface. A theoretical model was developed and revealed a good description of these phenomena. The theory attributes the superiority of nano-textured surfaces in comparison to the corresponding bare surfaces to the fact that the effective liquid temperature in liquid in the vicinity of such fluffy surfaces is higher than near the smooth surfaces. With the available experimental setups, it was possible to reach the critical heat flux (CHF) only with ethanol, and not water, as a working fluid. The experimental values of CHF on smooth and nano-textured surfaces were close and in good comparison with the existing theoretical result. However, the critical superheat temperature of nano-textured surfaces was about 10 °C lower than that on smooth surfaces. This result shows that nano-textured surfaces have significant benefits for surface cooling compared to that of smooth surfaces. Beyond CHF, in transitional boiling, heat removal from nano-textured surfaces was significantly more effective than from the smooth ones, since the former tend to prevent formation of vapor layer near the surface.

9. CRACKING OF CEILING TILE MATERIALS

9.1. Introduction

Ceiling tiles are commonly used inside buildings to improve acoustics and provide decoration, and for thermal insulation of rooms. The ceiling tiles USG makes are composed of perlite, USG mineral wool, corn starch, newsprint and water. To make ceiling tiles, these materials are mixed, and the initial raw material is a multiphase material with liquid enclosed in the pores and voids of a fluffy and flexible porous medium, which is kindred to foams in its geometric structure. This material undergoes several steps of processing continuously, which include free drainage, vacuum drainage, compaction and final drying. After that a dried sheet of ceiling material is cut into ceiling tiles. It was observed that while an "infinitely long" sheet of material passes on the conveyor belt over the vacuum section, cracking occurs perpendicular to the machine direction as shown in Fig. 9.1. This cracking is undesirable, weakens the material strength and can waste significant amounts of the formed ceiling tile sheet.

The aim of present Chapter is to elucidate the cracking mechanism and develop a proper physical model to rationalize and describe this phenomenon and search for ways of its elimination. The cracking experiments are described in section 9.2, and the corresponding theory is developed in section 9.3. The results are presented and discussed in section 9.4. Conclusions are drawn in section 9.5.



Fig. 9.1. Cracking of ceiling tile sheet over the vacuum section as it moves on the conveyor belt.

9.2. Experimental

In the production of USG ceiling tile material the slurry moves on the conveyor belt which has holes in it and water drains by gravity and vacuum as shown in Fig. 9.1. The present study is focused on the vacuum section where cracking occurs. The ceiling tile materials composed of perlite, USG mineral wool, corn starch, newsprint and water were supplied by USG in the form of slurry. A flocculant (OPTIMER 7128) was obtained from USG separately. A ceiling tile material batch of size of 500 g was mixed with 8 g of the flocculant. Then the mixed ceiling tile materials were deposited on a vacuum box which had a polycarbonate vacuum plate and brass screen mesh (mesh size 30x30 inch) on top of it, as depicted in Fig. 9.2. A flexible hose was connected between the vacuum box and a stainless steel pipe which collected water from the ceiling tile materials. To remove the collected water, a water drain line was installed. Vacuum (reduced) pressure which could

reach 580 mm Hg (the corresponding pressure at the sample bottom would be 180 mm Hg) was connected from the wall outlet (cf. the static setup shown in Fig. 9.2). Two types of vacuum plates were used to compare the effect of the vacuum openings on water drainage and cracking. One plate had circular holes and the other one had slots. Both vacuum plates had the same opening area of 28.3% of the total area (Fig. 9.3).



Fig. 9.2. Sketch of the static setup for vacuumization of USG ceiling tile materials.



Fig. 9.3. Drawing of vacuum plates (size units: mm). (a) Circular holes, (b) slots. Both plates possess the same opening area of 28.3% of the total area.

In the experiments the ceiling tile materials experienced free drainage for 30 s until the visible drainage of water had stopped. The amount of drained water removed during free drainage was measured by a scale. Then, the vacuum (reduced) pressure of 50-125 mm Hg (which means that the absolute pressure at the sample bottom was 635-710 mm Hg) was applied for 15 s from the wall outlet. The reason that the actual vacuum pressure was so small compared to the actual vacuum capacity was the following. When vacuum drainage occurs, a gap develops between the side of the material layer and the vacuum box walls, so that the magnitude of the vacuum pressure does not increase up to the full capacity. The drained water removed at the vacuumization stage was measured by the scale. After that, the slurry leftover which remained in the vacuum box was weighed by the scale. In the cases of reduced water content in the original ceiling tile materials, a part of the drained water was remixed with the leftover to make slurry, and the slurry underwent free and vacuum drainage.

The dried slurry leftover from the vacuum box was then carried to the three point bending setup for the bending test as shown in Fig. 9.4. On top of the material sample, a slender bar was placed to apply the force equally uniformly at the center line between two supports. A force transducer (Imada DS2-11) moved down to bend the ceiling tile materials by the force applied through the slender bar up to the sample cracking. The force magnitude was displayed by the force transducer in real time. The entire bending process and the force magnitude were simultaneously recorded by a CCD camera (Phantom Miro 4) from the side until the samples had started cracking. Several trials were done to ensure statistically reliable results.



Fig. 9.4. Sketch of the three point bending setup for USG ceiling tile materials.

Another bending setup was developed to measure the radius of curvature when the material starts cracking as depicted in Fig. 9.5. The material was buckled by two opposite compressive forces on the sides, and the top view images were recorded by the CCD camera (Phantom Miro 4). Simultaneously, the side view was recorded by another CCD camera (COHO 2222-2340).



Fig. 9.5. Sketch of the compressive buckling setup used to study cracking of the USG ceiling tile materials.

9.3. Theoretical Model

A plausible reason of cracking of the upper surface of ceiling tile material sheet moving over the vacuum section is that the upper surface bends and experiences significant stretching in the machine direction due to water suction into the vacuumization slot, as shown in Fig. 9.6.



Fig. 9.6. Sketch of ceiling tile material bending over a vacuumization slot while moving on the conveyor belt. h is the ceiling tile material height.

Consider vacuumization of ceiling tiles. From the plane theory of elasticity, stresses are fully determined via the Airy function U which satisfies the biharmonic equation (Muskhelishvili and Radok, 1975)

$$\nabla^4 \mathbf{U} = \mathbf{0} \tag{9.1}$$

In Cartesian coordinates x and y introduced in Fig. 9.7, Eq. (9.1) reads

$$\frac{\partial^4 U}{\partial x^4} + 2 \frac{\partial^4 U}{\partial x^2 \partial y^2} + \frac{\partial^4 U}{\partial y^4} = 0$$
(9.2)

The corresponding stresses are expressed though U as follows
$$\sigma_{xx} = \frac{\partial^2 U}{\partial y^2}, \ \sigma_{xy} = -\frac{\partial^2 U}{\partial x \partial y}, \ \sigma_{yy} = \frac{\partial^2 U}{\partial x^2}$$
(9.3)

The boundary conditions correspond to the distributed vacuum suction at the lower boundary of the sheet y=0 at x>0, and the compressive distributed reaction from the solid support at x<0 required to sustain equilibrium (cf. Figs. 9.7 and 9.8)

$$\sigma_{yy}\Big|_{y=0} = \sigma_{yy_0} x e^{-x^2} = \sigma_{yy_0} \frac{1}{2\sqrt{\pi}} \int_0^\infty k e^{-k^2/4} \sin(kx) dk$$
(9.4)

$$\sigma_{yy}\Big|_{y=1} = 0 \tag{9.5}$$

$$\sigma_{xy}\Big|_{y=0} = 0 \tag{9.6}$$

$$\sigma_{xy}\Big|_{y=1} = 0 \tag{9.7}$$

Here and hereinafter the coordinates are rendered dimensionless by the sheet thickness. In Eq. (9.4) and hereinafter presentations in the form of Fourier integral are used, with k being the wavenumber.



Fig. 9.7. The sketch of the ceiling tile materials sheet on the conveyor belt with x<0 being still over a solid support, and x>0 being over the vacuum slot. The applied normal stress at the lower surface is depicted by arrows.



Fig. 9.8. The graph corresponding to Eq. (9.4).

The solution of Eq. (9.2) is sought in the form of the following Fourier modes

$$\mathbf{U} = \mathbf{R}(\mathbf{y})\mathbf{e}^{\mathbf{i}\mathbf{k}\mathbf{x}} \tag{9.8}$$

which yields the following linear ordinary differential equation for the function R(y)

$$R^{iv} - 2k^2 R^{"} + k^4 R = 0$$
(9.9)

Its solutions is sought in the form

$$\mathbf{R} = \mathbf{e}^{\mathbf{p}\mathbf{y}} \tag{9.10}$$

Then

$$p^4 - 2k^2p^2 + k^4 = 0 (9.11)$$

which has two double roots $p = \pm k$.

Then the function $R_k(y)$ becomes

$$R_{k}(y) = A'_{k} \sinh(ky) + B'_{k} \cosh(ky) + C'_{k} y \sinh(ky) + D'_{k} y \cosh(ky)$$
(9.12)

In the special case of k=0, Eq. (9.9) becomes

$$\mathbf{R}^{\mathrm{iv}} = \mathbf{0} \tag{9.13}$$

Then

$$\mathbf{R}_{0}(\mathbf{y}) = \mathbf{M}_{0} + \mathbf{M}_{1}\mathbf{y} + \mathbf{M}_{2}\mathbf{y}^{2} + \mathbf{M}_{3}\mathbf{y}^{3}$$
(9.14)

where M_0 , M_1 , M_2 , and M_3 are constants.

If we include the other special cases, we arrive at the following general solution for U, i.e. using Eqs. (9.8), (9.12), (9.14), and the other special cases, the Airy function U can be written as

$$U = M_{o} + M_{1}y + M_{2}y^{2} + M_{3}y^{3} + N_{0} + N_{1}x + N_{2}x^{2} + N_{3}x^{3} + S_{1}xy + S_{2}xy^{2} + S_{3}yx^{2} + \int_{0}^{\infty} \left[(\sinh(ky) + B_{k}\cosh(ky) + C_{k}y\sinh(ky) + D_{k}y\cosh(ky)) \right]$$

$$(E_{k}\cos(kx) + P_{k}\sin(kx)) dk$$
(9.15)

where N_0 , N_1 , N_2 , N_3 , S_1 , S_2 , and S_3 are constants.

Since the stress $\sigma_{yy} = \partial^2 U / \partial x^2$, from Eq. (9.15) we can obtain

$$\sigma_{yy} = 2N_2 + 6N_3x + 2S_3y + \int_0^{\infty} \left[(-k^2)(\sinh(ky) + B_k \cosh(ky) + C_k y \sinh(ky) + D_k y \cosh(ky)) (E_k \cos(kx) + P_k \sin(kx)) \right] dk$$
(9.16)

The boundary condition Eq. (9.4) dictates

$$2N_{2} + 6N_{3}x + \int_{0}^{\infty} \left[B_{k}(-k^{2})(E_{k}\cos(kx) + P_{k}\sin(kx)) \right] dk =$$

$$\sigma_{yy_{0}} \frac{1}{2\sqrt{\pi}} \int_{0}^{\infty} ke^{-k^{2}/4}\sin(kx) dk$$
(9.17)

So $N_2=0$, $N_3=0$, $E_k=0$,

and

$$-B_{k}P_{k}k^{2} = \frac{\sigma_{yy_{0}}}{2\sqrt{\pi}}ke^{-k^{2}/4}$$
(9.18)

From another boundary condition, Eq. (9.5), we obtain

$$2S_{3} + \int_{0}^{\infty} (-k^{2}) [(\sinh(k) + B_{k} \cosh(k) + C_{k} \sinh(k) + D_{k} \cosh(k))P_{k} \sin(kx)] dk = 0$$
(9.19)

Therefore, S₃=0 and also

$$\sinh(k) + B_k \cosh(k) + C_k \sinh(k) + D_k \cosh(k) = 0$$
(9.20)

Since the stress $\sigma_{xy} = -\partial^2 U / \partial x \partial y$, from Eq. (9.15) we obtain

$$\sigma_{xy} = -(S_1 + S_2 y) - \int_0^{\infty} \left[(k \cosh(ky) + B_k k \sinh(ky) + C_k \sinh(ky) + C_k \sinh(ky) + D_k \cosh(ky) + D_k ky \sinh(ky)) k P_k \cos(kx) \right] dk$$
(9.21)

The boundary condition Eq. (9.6) dictates

$$-S_{1} - \int_{0}^{\infty} \left[(k + D_{k})kP_{k}\cos(kx) \right] dk = 0$$
(9.22)

Therefore, S₁=0 and

$$\mathbf{D}_{\mathbf{k}} = -\mathbf{k} \tag{9.23}$$

If we apply the boundary condition Eq. (9.7) and the expression (9.23) in Eq. (9.21), we obtain

$$-2S_{2} - \int_{0}^{\infty} \left[(B_{k}k \sinh(k) + C_{k} \sinh(k) + C_{k}k \cosh(k) - k^{2} \sinh(k)) + (P_{k}k \cosh(k) - k^{2} \sinh(k)) \right] dk = 0$$
(9.24)

Therefore, S₂=0 and

$$B_k k \sinh(k) + C_k (\sinh(k) + k \cosh(k)) = k^2 \sinh(k)$$
(9.25)

Substituting Eq. (9.23) into Eq. (9.20), we obtain

$$B_k \cosh(k) + C_k \sinh(k) = k \cosh(k) - \sinh(k)$$
(9.26)

From Eqs. (9.25) and (9.26) we find that

$$B_{k} = \frac{k^{2} - \sinh^{2}(k)}{k + \cosh(k)\sinh(k)}$$
(9.27)

and

$$C_{k} = \frac{k \sinh^{2}(k)}{k + \cosh(k)\sinh(k)}$$
(9.28)

Substituting Eqs. (9.27) and (9.28) into Eqs. (9.16) and (9.21), we obtain

$$\sigma_{yy} = \int_{0}^{\infty} (-k^{2}) P_{k} \sin(kx) \left[\sinh(ky) + \frac{k^{2} - \sinh^{2}(k)}{k + \cosh(k) \sinh(k)} \cosh(ky) + \frac{k^{2} \sinh^{2}(k)}{k + \cosh(k) \sinh(k)} y \sinh(ky) - ky \cosh(ky) \right] dk$$
(9.29)

and

$$\sigma_{xy} = -\int_{0}^{\infty} kP_k \cos(kx) \left[k \cosh(ky) + \frac{k^2 - \sinh^2(k)}{k + \cosh(k) \sinh(k)} k \sinh(ky) + \frac{k \sinh^2 k}{k + \cosh k \sinh k} (\sinh ky + ky \cosh ky) - k(\cosh ky + ky \sinh ky) \right] dk \qquad (9.30)$$

where $P_k = -\sigma_{yy_0}(k + \cosh k \sinh k) \exp(-k^2/4) / (2\sqrt{\pi}k(k^2 - \sinh^2 k)).$

From Eqs. (9.3) and (9.15) we can obtain $\,\sigma_{_{XX}}$

$$\sigma_{xx} = 2M_2 + 6M_3y + \int_0^{\infty} P_k \sin(kx) \frac{d^2}{dy^2} \left[\sinh(ky) + B_k \cosh(ky) + C_k y \sinh(ky) + D_k y \cosh(ky)\right] dk$$
(9.31)

Since the first two terms on the right side in Eq. (9.31) do not depend on the applied force, they should not be present, and thus $M_2 = M_3 = 0$

After a simple rearrangement, the stress $\,\sigma_{\scriptscriptstyle xx}\,$ is found as

$$\sigma_{xx} = \int_{0}^{\infty} P_k \sin(kx) \Big[(B_k k^2 + 2C_k K) \cosh(ky) + C_k k^2 y \sinh(ky) - k^2 \sinh(ky) - k^3 y \cosh(ky) \Big] dk$$
(9.32)

Then stresses $\,\sigma_{_{XX}}\,$ at the boundaries are

$$\sigma_{xx}|_{y=0} = \int_{0}^{\infty} P_k \sin(kx) (B_k k^2 + 2C_k k) dk$$
(9.33)

$$\sigma_{xx}|_{y=1} = \int_{0}^{\infty} P_{k} \sin(kx) \Big[(B_{k}k^{2} + 2C_{k}k) \cosh(k) + C_{k}k^{2} \sinh(k) - k^{3} \cosh(k) \Big] dk$$
(9.34)

Substituting Eqs. (9.18), (9.27), and (9.28) into Eqs. (9.33) and (9.34), we obtain

$$\sigma_{xx} \bigg|_{y=0} = -\frac{\sigma_{yy_0}}{2\sqrt{\pi}} \int_0^{\infty} e^{-k^2/4} \frac{k(k^2 + \sinh^2(k))}{k^2 - \sinh^2(k)} \sin(kx) dk$$
(9.35)

$$\sigma_{xx} \bigg|_{y=1} = \frac{\sigma_{yy_0}}{\sqrt{\pi}} \int_0^\infty e^{-k^2/4} \frac{k^2 \sinh k}{k^2 - \sinh^2 k} \sin kx dk$$
(9.36)

The values of both stresses can be evaluated by numerical integration and the results shown in Fig. 9.9. This means that the maximum value of σ_{xx} is 2.56 times larger than σ_{yy_0} .



Fig. 9.9. The values of Eqs. (9.35) and (9.36).

The stress of the top surface (y=1) under free drainage (x<0) is stretching and that under vacuum (x>0) is compressive as shown in Fig. 9.9. So cracking can occur at x<0 close to the boundary of vacuum section (x=0) close to the top surface.

9.4. Results and Discussion

9.4.1. Free and vacuum drainage at circular holes and slots

The experimental results show that there was no cracking developed during the free and vacuum drainage processes at the static setup irrespective of whether there were circular holes or slots in the vacuum plates. The % Dry which is defined in Eq. (9.37) was about 70% in the free drainage and increased to about 80% in the vacuum drainage as shown in TABLE 9.1 and Fig. 9.10. The corresponding % Water which is defined in Eq. (9.38) was about 88% in the free drainage and about 83% in the vacuum drainage as presented in TABLE 9.2 and Fig. 9.11. It is emphasized that removal of the additional amount of water at the vacuumization stage following the free drainage stage results in an increase of % Dry according to Eq. (9.37), and a decrease in % Water according to Eq. (9.38). These values are quite similar to USG's values. For example, in our case the value of the % Water after vacuum drainage is 83% and corresponding values in USG are 80-83%.

% Dry =
$$\frac{\text{Drained water (g)}}{\text{Initial water (g)}} \times 100$$
 (9.37)

$$\%Water = \frac{\text{Total solid leftover (water + solid) (g) - solid (g)}}{\text{Total solid leftover (water + solid) (g)}} \times 100$$
(9.38)

TABLE 9.1

% DRY IN FREE AND VACUUM DRAINAGE

	Circular holes						Slots					
No. of trial	1	2	3	4	5	Ave.	1	2	3	4	5	Ave.
Free drainage	69.0	74.1	72.7	71.5	59.6	69.4	71.6	72.8	74.0	70.6	61.1	70.0
Free + Vacuum drainage	81.3	81.9	79.1	76.2	83.9	80.5	82.1	77.6	82.8	80.2	79.7	80.5



Fig. 9.10. The graphic representation of the data in TABLE 9.1. (a) Free drainage, (b) free drainage followed by the vacuum drainage.

TABLE 9.2

%WATER (AS DEFINED BY USG) IN FREE AND VACUUM DRAINAGE

	Circular holes						slot					
No. of trial	1	2	3	4	5	Ave.	1	2	3	4	5	Ave.
Free drainage	88.6	86.7	87.2	87.3	90.9	88.1	87.5	87.4	86.9	87.9	90.2	88.0
Vacuum drainage	82.7	82.3	84.1	85.1	80.7	83.0	81.7	85.2	81.9	83.2	82.6	82.9



Fig. 9.11. The graphic representation of the data in TABLE 9.2. (a) Free drainage, (b) Free drainage followed by the vacuum drainage.

To investigate the %Water change by increasing vacuum pressure, a sealing lid was made and applied on top of the vacuum box and five trials of drainage experiment were done. This increased the vacuum pressure up to 580 mmHg which is the full capacity of UIC lab. The values of %Water after the high vacuum turned out to be almost the same as the low vacuum as presented in TABLE 9.3. Both %Water were approximately 81% and these values are the same as the values in USG.

	Vacuum							
No. of	pressure							USG's
trial	(mmHg)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average	value
% Water (1)								
(Low vacuum)	50-125	84.6	79.8	77.3	80.7	84.4	81.3	
% Water (2)								
(High vacuum)	480-580	84.3	79.8	76.9	80.0	84.1	81.0	80-83
%Water								
Diff. (1-2)		0.3	0	0.4	0.7	0.3	0.3	

%WATER (AS DEFINED BY USG) IN LOW AND HIGH VACUUM DRAINAGE

9.4.2. Bending experiment to characterize the material response of the partially dried materials

After the ceiling materials underwent the free and vacuum drainage, the solid leftover was installed in the bending setup (Fig. 9.4) to characterize its rheological properties. The materials with 10%, 20%, and 30% reduction in water content (accordingly, %Water is 96%, 95.6%, 95%, 94.4%) were studied. To that aim, a force F was applied normally to the sample by the force transducer and the corresponding deflection u at the center was measured as shown in Figs. 9.12 and 9.13. The material samples had sizes (the width and length; cf. Fig. 9.12) of 10 cm x 10 cm. The distance L between two supports was 5 cm. The mass of the solid leftover samples after vacuum drainage was about 125 g ± 10 g for all normal (96 % of Water), and reduced water

(95.6%, 95%, 94.4% of Water) cases. The sample thickness h was 1.3-1.5 cm as shown in TABLE 9.4.



Fig. 9.12. The sketch of force applied and deflection measured in pure bending of ceiling tile materials.



Fig. 9.13. The images of ceiling tile material samples subjected to pure bending. (a) The initial state. (b) The bent state at the moment when cracking starts at the bottom surface.

THE AVERAGE VALUES OF FOUR TRIALS OF STRENGTH-RELATED CHARACTERISTICS OF CEILING TILE MATERIALS WITH DIFFERENT WATER CONTENTS

Item	Unit	Normal water content (96%Water)	10% reduced water (95.6%Water)	20% reduced water (95% Water)	30% reduced water (94.4%Water)
Initial batch size (a)	g	500	452	404	356
Initial amount of water (b)	g	480	432	384	336
Initial %Water (=b/a)	%	96	95.6	95	94.4
%Water of vacuum drainage	%	83.5±1.5	83.7±1.6	83.8±1.1	83.5±0.9
Solid leftover	g	123±12.1	124±12.1	124±8.2	122±7.5
Solid leftover thickness	cm	1.48±0.02	1.44±0.05	1.37±0.06	1.35±0.05
Young's modulus	kPa	705±72	767±298	773±93	745±263
Yield stress	kPa	2.4±0.3	2.5±0.8	2.4±0.5	2.3±0.7
Strain at yield stress	%	0.34±0.02	0.33±0.04	0.31±0.05	0.31±0.03
Stress at cracking	kPa	12.5±1.3	11.1±1.7	11±1.3	10±1.2
Strain at cracking	%	10±2.0	7.8±3.2	7.4±1.3	6.6±3.3

The force-deflection dependences for different initial water contents are shown in Fig. 9.14. The force-deflection curves were transformed into the stress-strain dependences using the following formula. The strain at the top and bottom surfaces $(y=\pm h/2)$ achieves the maximum equal to

$$\varepsilon_{y=h/2} = \frac{h}{2R} \tag{9.39}$$

where h is the sample thickness, and R is the radius of curvature of the neutral surface $[u=R\{1-cos(arcsin(L/2R))\}]$. We assumed the sample is symmetric and subjected to pure bending only. Then the normal stress in the x-direction is given by

$$\sigma_{xx} = \frac{M_b h}{2I_{zz}}$$
(9.40)

where M_b is bending moment (=FL/4) and I_{zz} is the cross-sectional moment of inertia (=bh³/12).



Fig. 9.14. Force versus deformation of ceiling tile materials with different water contents. (a) Normal (96% Water), (b) 10% reduced water (95.6% Water), (c) 20% reduced water (95% Water), (d) 30% reduced water (94.4% Water).

The corresponding stress-strain dependences are shown in Fig. 9.15. The elastic limit which is defined as the maximum reversible deformation after unloading was measured in the bending experiment. It corresponded to the maximum deformation $u_{x=L/2}$ of 0.15 mm. In the range of the elastic response, Young's modulus was established (TABLE 9.4). When the plastic regime sets in, the yield stress and strain of the ceiling

tile materials were measured as shown in TABLE 9.4. The values of Young's modulus of the ceiling tile materials is approximately 700 kPa, the yield stress is 2.5 kPa, and the strain at yield is 0.3% regardless of the initial water content as shown in Figs. 9.16 and 9.17. The practically important factors are the stress and strain at cracking. Their values were measured as 10-12 kPa for the stress and 6-10% for the strain, respectively. This means that if a normal stress of 10 kPa is applied, the ceiling tile material will achieve a 6-10% strain and start cracking. To additionally verify a negligible effect of the initial (reduced) water content, the average values of stress and strain for each case were compared and shown to be quite similar to each other as is seen in Fig. 9.18.



Fig. 9.15. Stress-strain dependences of ceiling tile materials with different water contents. (a) Normal (96% Water), (b) 10% reduced water (95.6% Water), (c) 20% reduced water (95% Water), (d) 30% reduced water (94.4% Water).

The averaged stress-strain dependences of the solid samples with different initial water contents after free drainage and vacuumization are very similar to each other as shown in Fig. 9.18. The stress-strain dependences show that the ceiling tile materials possess a very small elastic zone (below $\varepsilon = 0.3$ %) followed by a subsequent nonlinear plastic-hardening zone as shown in Fig. 9.19. If the stress keeps increasing, cracking starts from the bottom surface.





Fig. 9.16. The average values of the strength characteristics of ceiling tile materials corresponding to TABLE 9.4. (a) Young's modulus. (b) Yield stress. (c) Strain at yield. (d) Stress at cracking. (e) Strain at cracking.



Fig. 9.17. The average values of the strength characteristics of ceiling tile materials corresponding to TABLE 9.4. (a) Young's modulus. (b) Yield stress. (c) Strain at yield. (d) Stress at cracking. (e) Strain at cracking. The horizontal axes correspond to the definition of water content as in Eq. (9.38).



Fig. 9.18. Averages stress-strain dependences of ceiling tile materials with different water contents.



Fig. 9.19. Average stress-strain dependence in bending of normal (96% Water) case. $\sigma_{\rm Y}$ is the yield stress.

9.4.3. Bending in compression: observations of cracking

To visualize and measure when the cracking exactly starts, another bending experiment was performed (Fig. 9.5). The results revealed that cracking occurred at approximately 10 cm radius of curvature found by AutoCAD software by image analysis (Fig. 9.20) and the corresponding strain was approximately 7% as shown in TABLE 9.5. This strain value is in good agreement with the results of the previous bending experiment described in subsection 9.4.2.



Fig. 9.20. The images of ceiling tile material sample as cracking starts in bending by compression. (a) Top view (the emerging crack is encircled by red line), (b) Side view. R is the radius of curvature of the neutral surface.

TABLE 9.5

No. of trial	1	2	3	4	5	Average
Sample thickness (cm)	1.3	1.25	1.45	1.52	1.35	1.37
Radius of curvature (cm)	6.9	9.4	7.9	12.7	13	10
Strain	0.094	0.066	0.092	0.06	0.052	0.069

MINIMUM RADIUS OF CURVATURE AT THE ONSET OF CRACKING

9.4.4. Comparison between the theoretical prediction and the results obtained on the USG pilot line

From the bending experiment we found the stress which causes cracking as 12.5 kPa [cf. TABLE 9.4 in section 9.4.2 and Fig. 9.21]. The theory developed in section 9.3 predicts that cracking can occur at x<0 close to the boundary of the vacuum section (x=0) at the top surface where the maximum stress is reached when vacuum is applied at the bottom surface as shown in Fig. 9.22. The theory also predicts that the maximum stress is 2.56 times higher than the value of the vacuum pressure [cf. Eq. (9.36)). This means the maximum stress reaches the critical cracking stress of $\sigma_{cracking} = 12.5$ kPa if the vacuum pressure is $\sigma_{yy_0} = 4.7$ kPa (35 mm Hg). In the experiments at the USG pilot line the vacuum pressure σ_{yy_0} use is in the range 13 mm Hg to 38 mm Hg at the low vacuum section. The predicted value of the vacuum pressure corresponding to cracking (35 mm Hg) belongs to the experimental interval. Therefore, the theoretical prediction is in good agreement with the experimental data. Also, cracking was, indeed, observed at the low

vacuum section of the USG pilot production line as shown in Fig. 9.23. Thus, the theoretical prediction shows a good agreement with the actual results.



Fig. 9.21. (a) Stress-strain curve and cracking stress, and (b) the image of ceiling tile material when it cracks.



Fig. 9.22. Sketch of ceiling tile material under vacuum applied at the bottom of the strip. Cracking occurs on the top surface of the ceiling tile material.



Fig. 9.23. Cracking location of ceiling tile material at the USG pilot production line. Cracking occurs on the top surface when the layer passes over the low vacuum section.

9.5. Conclusion

The experiments with ceiling tile materials showed that free drainage removes about 70 % of water. The subsequent vacuum-assisted drainage in a vacuum box revealed that the vacuum plates with circular holes and slots do not differ much in the drainage results. In both cases the additional 10% of water was removed in the vacuum box, raising the total amount of water removed up to about 80%. No cracking was found in the experiments in the static vacuum-box setup. The tensile properties were measured using the bending setup and revealed the following values. Young's modulus corresponding to the elastic response in the elastic domain (strains below 0.3 %) was found to be 700 kPa, the yield stress 2.4 kPa, the cracking stress and strain 12.5 kPa and 6-10%, respectively. These parameter values practically did not change much between the normal (96% Water) to 30% reduced water (94.4% Water) cases. The compressive bending setup was employed to observe crack formation. A plausible cause of cracking can be attributed to

bending of the sample upper surface as the sample starts diminishing as a result of water removal through the vacuum holes or slots. The hypothesis was tested and found that the cracking would occur at the low vacuum section on the top surface of the ceiling material on the conveyor. This prediction is in agreement with the cracking observed at the USG pilot production line, where cracking occurs at the upper surface of the ceiling material layer when it is moving over the low vacuum section on the conveyor belt.

10. CONCLUSION

In this thesis transport phenomena in several kindred classes of multi-phase materials (slurries, foams, boiling bubbly media, and pulps) were studied experimentally and theoretically having in mind a wide scope of potential applications. These include the construction industry, cosmetics and food industry. The range of potential applications also incorporates cooling of electronics, boilers, nuclear and chemical reactors, and refrigeration systems.

Rheological behavior of gypsum slurries with foam was explored experimentally. The results reveal shear-thinning behavior with the rheological parameters strongly dependent on foam content. Rheological parameters of the power-law Ostwald-de Waele rheological constitutive equation n and K (the exponent and consistency index) were established as the functions of the foam content. These results can be applied in numerical calculations of mixing foam with gypsum slurries for the real gypsum wallboard production lines in Unites States Gypsum (USG) Company.

A novel theory for gravitational foam drainage was developed in the frame of the consolidation theory. The results showed that there are two different drainage regimes and their rates of drainage differ by a factor of 2. This result was corroborated in the experimental PhD work of D. Pelot.

The foam thread theory was developed and predicted catenoidal shape when thread length is smaller than two-thirds of its diameter. The experiments showed that relatively stable foam threads can be achieved with 8% Pantene concentration in water. The addition of 0.05 g of PEO enhances thread stability even more. The increase in the

Pantene concentration and the addition of PEO result in progressively longer-living quasi-steady foam threads. The theoretically predicted maximum length of the steady thread is in a good agreement with the experimental observations. The theoretically predicted steady-state thread profiles agree with the experimental data with the maximum difference being in the range of 3-11%.

In the part devoted to pool boiling on nano-textured copper surfaces coated with copper nanofibers, it was shown that a 6-8 times higher heat flux and heat transfer coefficient in nucleate boiling of ethanol can be achieved in comparison with those of the corresponding bare copper surfaces. For the nucleate boiling of water, the heat flux and heat transfer coefficient on nano-textured surfaces were 3-5 times higher than those of the corresponding bare copper surface. A theoretical model was developed and revealed an explanation and an accurate description of these phenomena. The theory attributes the superiority of nano-textured surfaces in comparison to the corresponding bare surfaces to the fact that the effective liquid temperature in liquid in the vicinity of such fluffy surfaces is higher than near the smooth surfaces. With the available experimental setups, it was possible to reach the critical heat flux (CHF) only with ethanol, and not water, as a working fluid. The experimental values of CHF on smooth and nano-textured surfaces were close and in good agreement with the existing theoretical result. However, the critical superheat temperature of nano-textured surfaces was about 10 °C lower than that of the smooth surfaces. This result shows that nano-textured surfaces reveal significant benefits in comparison with smooth surfaces. Beyond CHF, in transitional boiling, heat removal from nano-textured surfaces was significantly more effective than from the smooth ones, since the former tend to prevent formation of a vapor layer near the surface.

Cracking of pulp-like USG ceiling tile materials was studied experimentally and theoretically. The plausible causes of cracking were found to be related to the slope changes as the layer thickness diminishes at the border of the free and vacuum drainage sections of the conveyor belt. This causes stretching stress in layer cross-section near the surface, with an especially high value at the top surface of the material. Such stretching stress results in cracking. The stress-strain relation of ceiling tile materials was measured in three separate bending experiments, which also established values of Young's modus, the yield stress, and material strength. The theoretical results were corroborated by the experimental data acquired for the USG pulp and good agreement of the results was demonstrated.

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