

Influence of Suspension Properties on the Fabrication of SiC Ceramic Products by Slip Casting

**Påvirkning av suspensjonsegenskaper på
fabrikasjon av SiC keramiske produkter ved
slikkerstøping**

Bachelor Thesis

Project no: IMA-B-19-2020

Delivery date: 03 June 2020

Confidential/Open: Open

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Preface

This bachelor project is completed during the spring of 2020 at the Institute of Materials Technology, Norwegian University of Science and Technology (NTNU) in the spring of 2020.

The project is provided by Washington Mills AS, Orkanger. The main purpose of this assignment is to investigate the effect of powder properties on slip quality for the production of slip casting green bodies for recrystallized silicon carbide components. This topic was chosen because of my interest in ceramic production and the future development prospects.

I would like to extend my gratitude to my patient supervisors Kjersti Kleveland from NTNU and Anne Marie M. Moe from Washington Mills AS. They have given me excellent support and guidance over the whole project period.

Lastly, I would like to thank Paul Ulvan for his detailed manufacture explanation and Bjørn Almi for his proper operating in the laboratory.

Orkanger, May 2020

Abstract

The main content of this thesis is the influence of slip properties on the fabrication of silicon carbide products by slip casting. Silicon carbide ceramics have high temperature stability, high mechanical strength and high thermal conductivity, and its application range is becoming more and more extensive. For satisfying the needs of industrial production, slip casting is used to produce silicon carbide products required by the market. This casting technique requires the preparation of silicon carbide slip with high solid content, low viscosity, and good stability.

In order to research the conductivity, pH and viscosity of silicon carbide ceramic slip, firstly, the properties of silicon carbide slip were studied. Such as rheological properties changing the flow and deformation behavior of slip, dispersion mechanism of dispersant preventing particles approaching, and the effects of the slip properties on green body which adjust the pH value in the slip can adjust the electric charge on the particles surface, so the electric charge increases, the electric double layer repulsion increases, and a stable slip state is achieved.

Basic theory of controlling stabilized ceramic slip was obtained. Then, some experiments were done to analyze the factors which affect the viscosity of silicon carbide slip. Such as conductivity, pH value, dispersants and solid content. However, because the experimental work began in the period of the Covid-19 closure that the original planned slip casting was cancelled, and the laboratory was closed as well. So only the results from completed experiments and the lab. data from Washington Mills AS was analyzed in this paper.

The experimental results showed that the acid washing process of SiC fine powder has a significant effect on the conductivity and pH of silicon carbide slip. The silicon carbide slip with unacid-treated powder has high conductivity because of higher iron content. The silicon carbide slip with acid-treated powder has relatively lower conductivity. The conductivity was decreased from 40.5 $\mu\text{S}/\text{cm}$ to 13.9 $\mu\text{S}/\text{cm}$ and pH from 6.41 to 4.44. The slip stability was analyzed by viscosity measurement as a function of time. The best conditions of stable slip were selected on the basis of low viscosity with high solid content. It was observed that 0.012 wt% of AMP-dispersant (2-amino-2-methyl-1-propanol) resulted lowest viscosity of the slip with 80 wt% solid content.

At the end of this paper, further work was also suggested. Such as the re-measure the viscosity to observe the settlement and solidification of the slip. Study the rheological properties of SiC slip to confirm whether the fluid is Bingham plastic fluid or other types of fluid. Discover new methods to measure shear stress and shear rate of viscosity measurement and recommend using disc-spindles (spindle nr. 62 and 63 of Brookfield DV-II+ viscometer) instead of cylinder-spindles (spindle nr. 61 and 64). Perform slip casting process on silicon carbide

slip to study the slip effect on the green bodies. And also check different type of dispersants with different amount to prepare silicon carbide slip with good stability and high solid content.

Sammendrag

Hovedinnholdet i denne oppgaven er påvirkningen av suspensjonsegenskaper på fabrikasjonen av silisiumkarbidprodukter ved slikkerstøping. Silisiumkarbid keramikk har høy temperaturstabilitet, høy mekanisk styrke og god varmeledningsevne, og anvendelsesområdet blir mer og mer omfattende. For å tilfredsstille behovene i industriproduksjon, brukes slikkerstøping til å produsere silisiumkarbidprodukter som markedet krever. Denne støpingsteknikken krever fremstilling av silisiumkarbid slikker med høyt solidinnhold, lav viskositet og god stabilitet.

For å undersøke konduktiviteten, pH og viskositeten til silisiumkarbid suspensjon, ble det først studert egenskapene til silisiumkarbid slikker. Så som reologiske egenskaper som endrer flyt og deformasjonsatferd for suspensjon, dispersjonsmekanisme for dispergeringsmiddel som forhindrer at partikler nærmer seg, og virkningene av slikker egenskapene på grønnkropper som justerer pH-verdien i slikker kan justere den elektriske ladningen på partikkeloverflaten, så den elektriske ladningen øker, den elektriske dobbel-lag frastøtningen øker, og en stabil slikker tilstand oppnås.

Grunnleggende teori for kontroll av stabilisert slikker ble oppnådd. Deretter ble noen eksperimenter utført for å analysere faktorene som påvirker viskositeten til silisiumkarbid slikker, så som konduktivitet, pH-verdi, dispergeringsmidler og solid innhold. På grunn av det eksperimentelle arbeidet begynte i perioden med Covid-19 stengning, så ble imidlertid den opprinnelige planlagte slip casting kansellert, og laboratoriet ble også stengt. Så kun resultatene fra gjennomførte eksperimenter og lab fata fra Washington Mills AS ble analysert i denne oppgaven.

De eksperimentelle resultatene viste at syrevaskingsprosessen til silisium karbid fint pulver har en betydelig innvirkning på pH og konduktivitet av silisium karbid slikker. Slikker med pulver som var ikke syre-vasket har høy konduktivitet på grunn av høyt jerninnhold, men slikker med syre-vasket pulver har relativt lavere ledningsevne. Syre vasking prosessen gjør at konduktiviteten ble redusert fra 40,5 $\mu\text{S}/\text{cm}$ til 13,9 $\mu\text{S}/\text{cm}$ og pH fra 6,41 til 4,44. Slikkerstabiliteten ble analysert ved viskositetsmåling som en funksjon av tiden. De beste betingelsene for stabil slikker ble valgt på basis av lav viskositet med høyt solid innhold. Det ble observert at 0,012 wt% med AMP-dispergeringsmiddel (2-amino-2-methyl-1-propanol) resulterte i laveste viskositet av slikker med 80 wt% solid innhold.

På slutten av denne oppgaven ble det også foreslått videre arbeid. Som for eksempel å måle viskositetsprøvene til å observere avvikling og størkning av slikker.

Studer de reologiske egenskapene til silisium karbid slikker til å bekrefte om slikker er Bingham plast fluid ellers annen type fluid. Oppdager nye metoder for å måle skjærspenning og skjærhastighet til viskositetsmåling, anbefaler å bruke skive-spindler (spindel no. 62 og 63 fra Brookfield DV-II+ viskometer) istedenfor sylindrisk spindler (spindel no. 61 og 64). Utfør slikkerstøping prosess på silisium karbid slikker, og studer slikkers effekten på grønnkropper. Finn ut ulike dispergeringsmidler med forskjellig mengde for å tilberede slikker med god stabilitet og høyt solid innhold i slikker .

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1 Introduction

Silicon carbide ceramics are compounds with extremely strong covalent bonds which maintain high bonding strength at high temperatures, and the coefficient of thermal expansion is small, and the corrosion resistance is excellent. Therefore, the silicon carbide ceramics material is considered to be one of the most promising materials as high-temperature structural component.

The application range of silicon carbide ceramics is getting wider and wider. Not only as traditional abrasives, heating elements and general refractory materials, but also widely used in the ceramic industry as sagger, grids, flameproof plates. And other structural components used under harsh conditions as wear-resistant sealing rings. The demand for silicon carbide is increasing. It can be used as a mechanical measuring gauge, precision bearing, compressor cylinders and pistons, anti-wear seals. Especially the seals used for slurry pumps with solid particles flushing, shows superior characteristics than the cemented carbide materials. At relatively high operating temperatures (above 1000°C), it can also be used as rotors, nozzles, and burners for high-temperature gas turbines [1].



Figure 1: Silicon carbide [2]

Pure silicon carbide is an insulator. When impurities are present, the resistivity drops to a very low value, the conductivity increases, and it has a negative charged temperature coefficient. It can be used as a non-linear resistance and high temperature heating element [3]. In summary, silicon carbide materials are widely used and are more and more attractive in the market. The traditional dry-pressing has been difficult to satisfy the requirements of modern practical and industrialization, so the development of silicon carbide slip casting has a strong

practical significance. Moreover, the process equipment for slip casting has characteristic of low requirements, low cost and simple operation [1]. Silicon carbide is different from oxide ceramics (such as alumina). It is difficult to obtain a slip with good dispersibility and high suspend ability. This assignment mainly studies how to improve the properties of silicon carbide slip. Conductivity measurement shows the purity of powder which means the higher purity of powder, the lower impurity that dissolves in the water. And viscosity affect the slip quality in the casting process. Therefore, it is important to find the best ratio of liquid, powder and dispersant to prepare low liquid content, as much high solid content with low viscosity as possible. This is determined by several slip mixtures and viscosity measurement with different proportions of slips [4].

2 Theory

Since the middle of this century, high-performance fine ceramics have gradually developed. Especially in the past two decades, oxide ceramics, non-oxide ceramics and ceramic matrix composites were developed rapidly, with its excellent properties such as high-temperature resistance, high strength, wear resistance, and corrosion resistance, ceramics have been increasingly used in aerospace, energy, chemical, mechanical, biological sciences and other fields. Silicon carbide ceramics have high mechanical strength, high temperature resistance, wear resistance, corrosion resistance, small thermal expansion coefficient, high thermal conductivity, etc., and are often used in the manufacture of combustion chambers, high temperature exhaust devices, temperature resistant patches, aircraft engine components, chemical reaction vessel tubes and other mechanical components under severe conditions. Silicon carbide is a widely used advanced engineering material, and the products have a broad market and application areas to be developed in the fields of energy, metallurgy, machinery, building materials and chemicals. To reach the requirements of high and new ceramic technology, it is necessary to produce various silicon carbide products with different levels and different properties [5].

2.1 General ceramic manufacturing process

Since the middle of this century, high-performance fine ceramics have gradually developed. Especially in the past two decades, oxide ceramics, non-oxide ceramics and ceramic matrix composites were developed rapidly, with its excellent properties such as high-temperature resistance, high strength, wear resistance, and corrosion resistance, ceramics have been increasingly used in aerospace, energy, chemical, mechanical, biological sciences and other fields. The manufacturing of ceramics can be created by many methods, and the objective is to produce a solid product with the desired shape and form with the right raw material. As the first attempt, the main manufacturing process divides into five groups, depending on the sequence of the production process (*Figure 2*) [6].

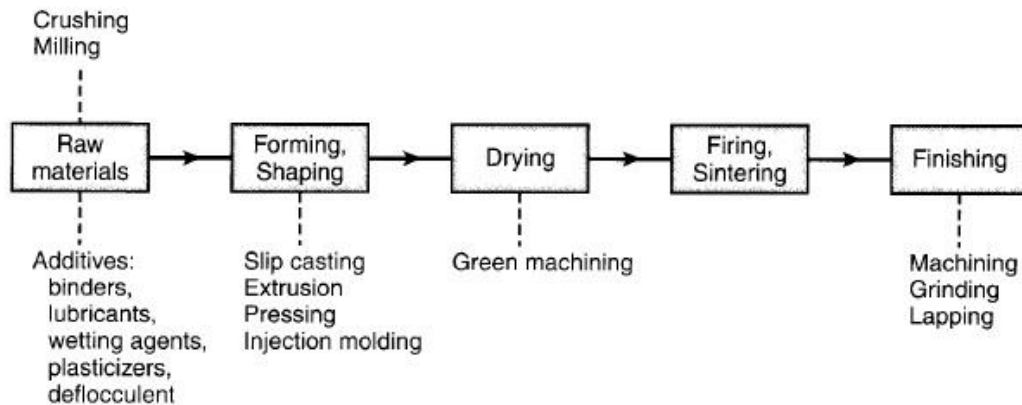


Figure 2: Ceramic forming process [7]

However, the industrialization and application of ceramics also face some serious problems such as the manufacturing cost is high, and the brittleness is large. To effectively solve these problems, ceramic material scientists focused on the research of ceramic material preparation technology, such as additives in forming process, slip properties research and sintering process [7].

2.2 Fabrication and production of Silicon Carbide

2.2.1 Silicon carbide

Silicon Carbide, also known as Carborite, Gold Sand or Refractory Sand. Chemical formula is SiC. SiC is a kind of refractory produced at high temperature in resistance furnace using quartz sand, petroleum coke and wood chips as raw materials. SiC presents in nature as a rare mineral which is in moissanite. SiC are also the most widely used and economical refractory raw materials. Industrial production of SiC is divided into three types (*Figure 3*). Black SiC, green SiC (both are hexagonal crystals which are α -SiC [8]), and a lower quality of metallurgical SiC.



Figure 3: Black and green SiC powder [9]

The black SiC is about 95% of all SiC production, the black SiC toughness is higher than the green SiC. Most of them are process materials with low tensile strength, for example: non-ferrous metals, glass, stone, refractories, cast iron and ceramics. The green SiC has 97% of pure SiC with a good sharpening property [10]. There are two main types of SiC crystals, hexagonal or rhombohedral α -SiC and cubic β -SiC. Many different α -SiC variants are formed due to the different stacking order of carbon and silicon atoms in their crystal structure. At 2100°C or higher, β -SiC is converted to α -SiC. The production method of industrial SiC is refined using high-quality quartz sand and petroleum coke in a resistance furnace. Through grinding, acid-base washing, magnetic separation, sieving or water selection, the obtained SiC block can be made into products of various particle sizes. From SiC binary diagram (*Figure 4*) that SiC does not melt at low temperatures. It begins to decompose into silicon liquid and carbon when the temperature is above 2830°C. In fact, SiC begins to decompose from 1500-

2000°C, and has decomposed significantly above 2830°C. The 50 at% carbon concentration is the separatrix between α -SiC and β -SiC. α -SiC below 50 at% and β -SiC above 50 at%. Also α -SiC forms when the temperature is greater than 1700°C and β -SiC forms when the temperature is lower than 1700°C [11].

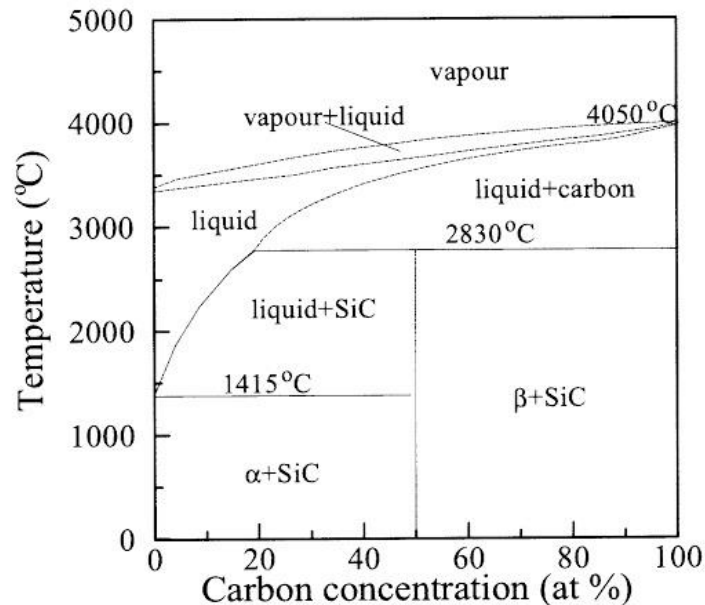


Figure 4 The binary diagram of Si-C [12]

The α -SiC commercially synthesized in large quantities by the Acheson method. This fascinating process looks rough, but it is low cost while producing both low-grade SiC for abrasives and high-grade SiC for electrical applications. The Acheson process (Figure 5) essentially a resistance furnace with a carbonaceous material as the core body heats the mixture of quartz (SiO_2) and carbon to produce silicon carbide [13].

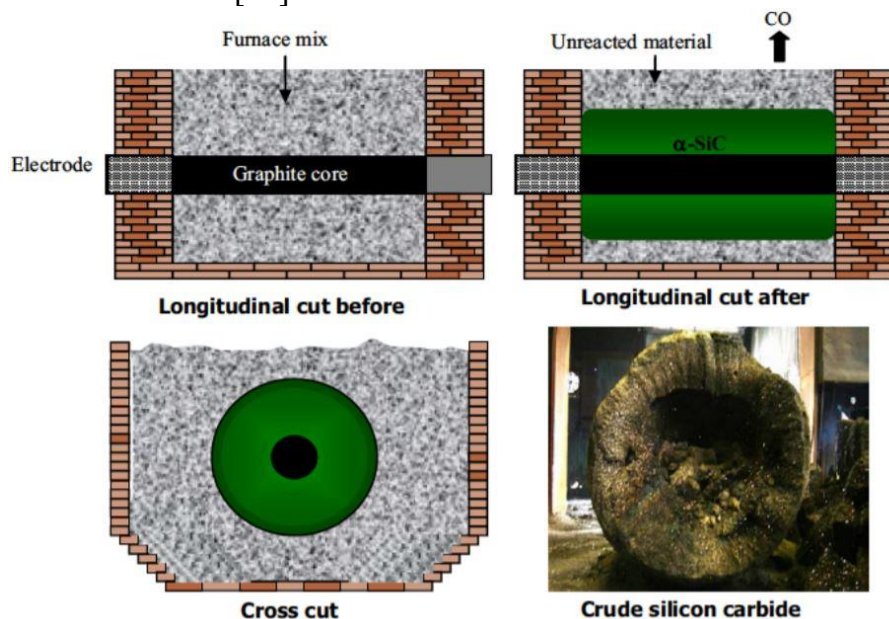
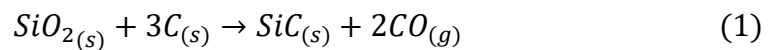


Figure 5: The Acheson furnace and the crude silicon carbide product [13]

2.2.2 Silicon carbide powder production

Washington Mills AS uses the traditional Acheson process to produce SiC powder. The process starts at an oven hall. Mix carbon (petrol coke, also cole from mines can be used) and quartz sand and lay the bed of this mixture before passing the carbon core across the bed. Add a new layer of carbon and quartz sand mixture on it. Now this is similar to a rectangular pencil, where the graphite core is the core of the pencil and the wood is a mixture of quartz sand and carbon. The power supply runs on this carbon core, causing high temperature and gas-phase reaction, in which silicon carbide crystals grow:



The size of the oven is different, and usually a silicon carbide crude like a roll cake. The diameter can be 1 to 1.5 m and the length are a few meters. Silicon carbide crude is separated from unreacted reactants which can be reused. Silicon carbide crystals are then crushed down with a jaw crusher, grinders and roller crushers down to a size about 500 μ m/F (also called 500/F). In principle, there are term after each crushing step. Those do not pass the screening need to go through the crushing step again. The silicon carbide powder with the size 500 μ m/F, goes into a jet mill which crushes it down to size about 200 μ m/F. This powder goes to a mechanical production screen, where the composition of the fabrics is desired quality as 100 μ m/F. The 100 μ m/F silicon carbide size is relatively coarse fraction of the finished products handled by Washington Mills AS, Orkanger. The 100/F silicon carbide can be crushed down to finer fractions. FPG7 is such a nice fraction. Crushing down to this fraction can occur through a jet milling or ball milling. The jet milling is very energy-intensive in comparison with ball milling, but at the same time has the advantage of being pollution-free between the particle-particle crushing [14].

2.2.3 Acid washing process

In the industrial production of SiC fine powder, the raw materials inevitably contain impurities such as Fe, free Si, SiO₂, and organic silicon impurities. For improve the purity of SiC powder, remove as much as possible iron from the SiC powder. The raw powders were washed with an acidic solution. In the washing process, SiC is mixed with water to form a thin liquid slip. the mixture is added to sulfuric acid (H₂SO₄) and the washing takes place under continuous stirring for a few hours. Sulfuric acid is effective for settling powder. The powder settles for about 24 hours when the agitator stops. The peak phase with dissolved ions can then be sucked out and new water added before dewatering and rinsing in the filter press. The powder is dried and packed [15].

2.2.4 Classification of silicon carbide powder

The silicon carbide powder products from Washington Mills are mainly classified according to applications of the powder. CARBOREX are alpha, high quality, silicon carbide macro grits, micro grits, powders and sub-micron powders for general and abrasives applications such as grinding, cutting, shaping, honing and polishing applications, also for advanced or technical ceramics. CARBOREX NG offers exceptional flow properties and stable oxidation properties. CARBOREX No.1 RF for refractory applications such as furnace and incinerator linings, kiln furniture, fire bricks, cement and electric heating elements. CARBOREX FPG7 and 100/F for recrystallized and nitride bonded ceramics. CARBOREX MA for metallurgical applications [16].

2.3 Slip casting of SiC ceramics

Casting Methods is more practical. It has many advantages, such as simple equipment, uniform distribution of green body, fewer defects and more suitable for the production of complex parts [17].

Slip casting (*Figure 6*) is a common method used to shape silicon carbide ceramic products. The suitable amount of additives (such as wetting agents, dispersing agent or deflocculant) to dissolve the raw silicon carbide powder in the solution with a stable condition and prepared a slip. The slip is poured into the plaster mold which has the character of absorbing moisture from slip. After the water is absorbed by plaster mold, a uniform slip layer with a certain thickness is formed (formation of slip casting as shown in *Figure 6*). During the dehydration and draining process without heat, a green body with a certain hardness is formed at this time and drained the slip out. The green body goes through the partial drying and separated from a plaster mold. The heat is applied after the green body is shaped and goes through a sintering process. [18]

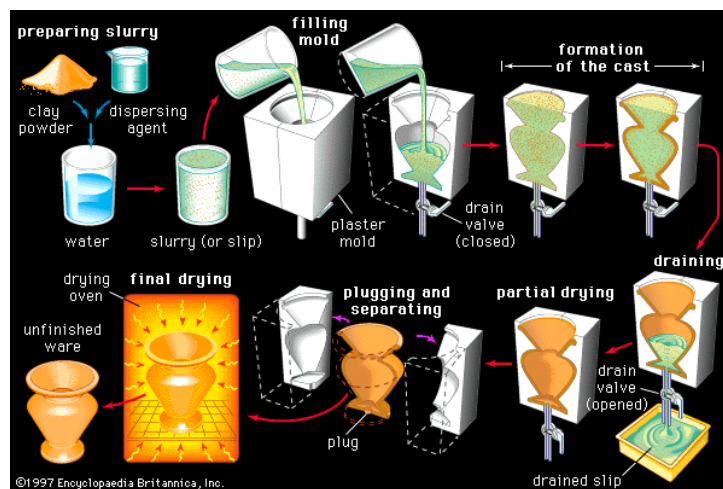


Figure 6 slip casting process [18]

The slip casting has the characteristics of strong practicability, simple operation, low cost and uniform particle distribution for silicon carbide ceramic products. The key to casting is the preparation of silicon carbide slip with high solid content (powder content), low viscosity, good flowability and stability.

The high solid content helps to increase the density of the product and reduce crack and deformation during sintering. Low viscosity is beneficial to improve the flowability of the slurry and eliminate air bubbles in the slip. In order to improve the dispersion behavior of powder in the liquid medium (water) and obtain a ceramic slip with better flowability, a dispersant is usually added to the slip. Therefore, it is necessary to have research on the properties of ceramic slip [5].

2.4 Properties of silicon carbide slip

2.4.1 Rheological properties

The flow and deformation behavior of slurry are called rheological property. The rheological properties of substance are very practical. The most commonly used rheological property in slip is viscosity. Viscosity represents the amount of internal friction when a substance flow. When it flows, a shear rate (velocity gradient) will be formed inside the liquid and producing the flow resistance. The amount of this resistance is called shear stress and is related to the shear rate. Newton's law of viscosity states that the shear stress in a flowing fluid is directly proportional to the rate of shear strain [19].

$$\tau = \mu \cdot \frac{du}{dy} \quad (2)$$

Where τ (Pa) is the shear stress, μ (Pa · s) is the viscosity and $\frac{du}{dy}$ (s^{-1}) is the shear rate.

This formula signified the fluids which follow Newton's law of viscosity are called Newtonian fluids. Newtonian fluids characteristic is that the viscosity is only related to temperature, not affected by the shear rate. The shear stress is proportional to shear rate. Non-Newtonian fluid: does not match Newton's formula, which is means the shear stress and shear rate are not proportional. For the viscosity of the slip, the influencing factors are as follows: the particle shapes, temperature, the electric charge and the concentration of the dispersed phase. For slip liquids, their rheological properties are much more complicated. Plotting shear rate against shear stress gives the flow curve shown in *Figure 7*, which represents the rheological properties of the flow system [19].

For Newtonian fluid, the rheological curve is a straight line passing through the origin. Which is means the slip can flow under any small external force. Plastic fluid, also called Bingham fluid. The rheological curve is also a straight line, but it does not pass through the origin, but intersects the shear stress axis at τ_y point. Only when $\tau > \tau_y$, the fluid flows. τ_y is called the yield stress. For pseudoplastic fluids, there is no yield stress. The reduced value between shear stress and shear rate, decreases with increasing shear rate. In other words, the viscosity becomes smaller as the intensive stirring increases. The faster stirred, the thinner fluid appears. The dilatant fluid, the rheological curve passes through the origin, but opposite from the pseudoplastic fluid. The viscosity of the dilatant fluid increases with the increased shear rate, which means the faster the fluid stirred, the thicker it appears. [19]

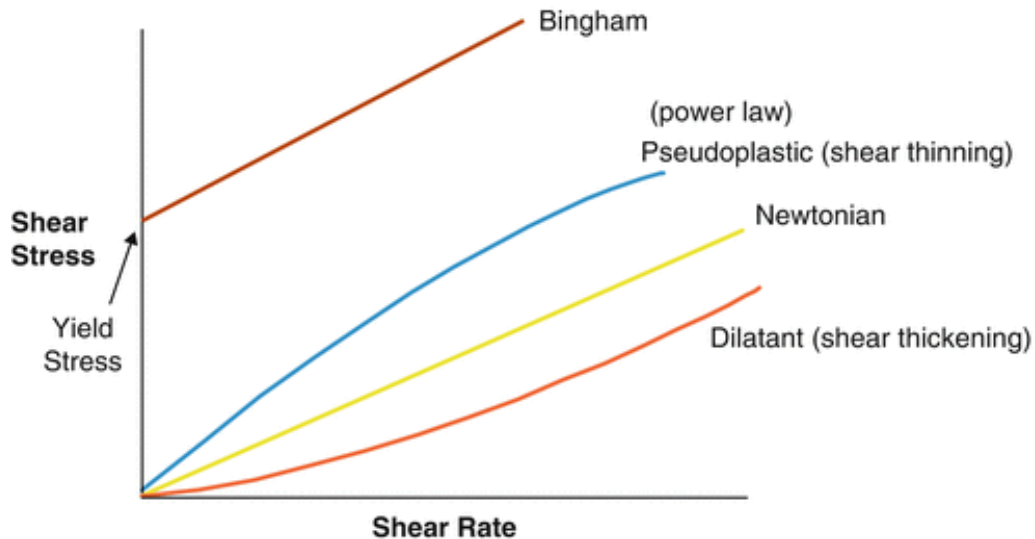


Figure 7 Basic types of fluids [19]

SiC ceramic slip is generally Bingham fluid [20]. The slip which contains larger particle size and higher solid phase. The Bingham slip transformed to the dilatant character, shear thickening. Shear thickening occurs mainly due to the slurry transformation from orderly liquid to disorderly liquid. When the shear rate is reduced, the shear stress is small, and particles are surrounded by electrostatic repulsion. When the shear rate increases, the shear stress will exceed the electrostatic repulsion between particles and causing the particles deviated from the equilibrium position. As a result, the particles are in disorder and the viscosity of the slurry increases. This theory predicts that when the solid phase volume is high, the solid phase particle size is large, the particle surface potential is low, shear thickening is likely to occur. However, the order to disorder slurry transition is difficult to explain the reverse process of shear thickening (that is when the shear thickening occurs, the shear rate decreases, the viscosity of the slurry decreases, and the state before the shear thickening is restored.). Another view is that the shear thickening is due to the interaction of the repulsive barriers of the particles. When the shear rate increases, the shear stress is large, making the particles closer to each other, but due to the interaction of the repulsive potential energy between the particles, a large repulsive force is generated between the particles, which increased slurry viscosity. In this view, shows the smaller the particle surface potential, the better to avoid shearing thickening. A surface potential is too large, will increase the range of repulsive potential energy and make shear-thickening more likely to occur. The shear thickening of ceramic slurry has a big influence on the slurry mixing, ball milling process and slip casting etc. [21]

2.4.2 Dispersion mechanism of dispersant

The dispersion of ceramic slurry generally includes three processes: wetting, mechanical crushing and stable dispersion. Wetting is the process where the particle-particle interface is replaced by the interface between the particle and the dispersant (or other organic additives such as solvents). Mechanical crushing is the process of refining large particles, deagglomerating on the aggregates, and moistening and wrapping them again. Stable dispersion is a process which slip particle can shield the Van der Waals forces through electrostatic repulsion and air repulsive forces, so that the particles are no longer aggregated. The dispersant usage is a very important for slurry. Slip casting requires the ceramic slip to be completely stable and dispersed. The dispersant in the slip will interact with the surface of the ceramic particles to prevent the occurrence of agglomeration between particles. When the radius of the ceramic particles is R (cm), in the dispersion medium with dynamic viscosity μ ($\text{dyne} \cdot (\text{s}/\text{cm}^2) = (\text{g} \cdot \text{cm}/\text{s}^2) \cdot (\text{s}/\text{cm}^2)$), particles will sink due to gravity g (cm/s^2), and its falling velocity v (cm/s) can be expressed as Stokes formula:

$$v = \frac{2gR^2(\rho_p - \rho_f)}{9\mu} \quad (3)$$

Where $\rho_p > \rho_f$ when it is falling. ρ_p (g/cm^3) is the density of the particles, and ρ_f (g/cm^3) is the fluid density. This is the falling velocity of the particles, but the particles also have Brownian motion. The smaller the particles, the more obvious the Brownian motion. Therefore, if the agglomeration between the particles can be restrained, the particles can be dispersed in water steady without being affected by particles diameter R [20].

There are two main effects for the dispersant in the slip: Electrostatic effect and steric effect (*Figure 8*). Electrostatic effect is the ionic dispersant adsorbs on the surface of the particles, increasing the repulsive force of the electric double layer and keeping the particles in a stable slip state in the dispersion medium, as shown in *Figure 8a*. Steric effect, the high uncharged polymer is adsorbed on the surface of the particles, and the solvated chain extends into the dispersion medium and spreads out, preventing the particles from approaching each other, as shown in *Figure 8b*. The dispersed particles are always in a collision state, so the dispersant must have sufficient adhesion to avoid desorption. And there must be sufficient concentration to prevent coalescence of particles caused by collision [22].

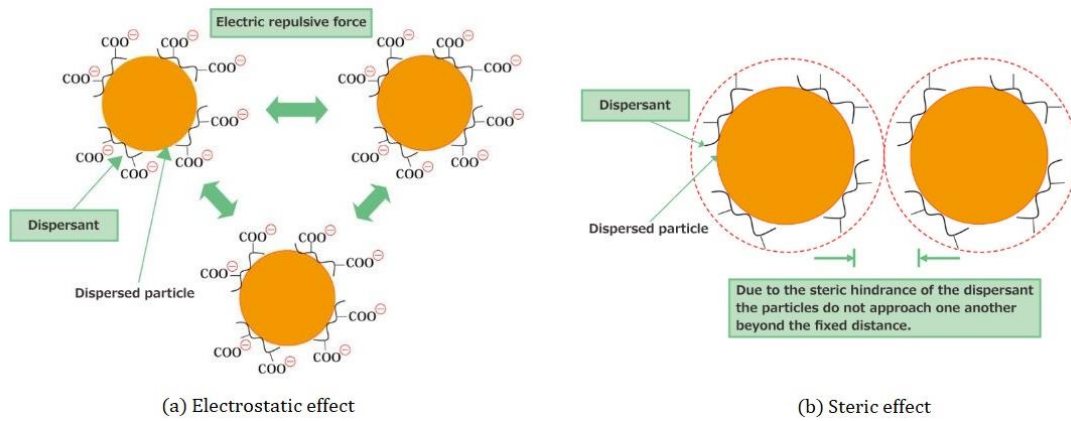


Figure 8: The function of dispersant [22]

The dispersant which used in this thesis is AMP-dispersant (2-amino-2-methyl-1-propanol). AMP-dispersant utilizes the principles of electrostatic effect. The chemical structure as shown in *Figure 9*. When the hydrophilic OH-group in the AMP molecule is adsorbed on the particle surface, and NH^3+ charge is formed on the surface [23].

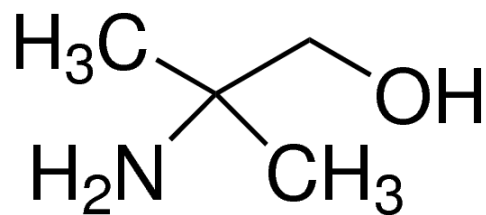


Figure 9: The structure of AMP-dispersant [24].

2.4.3 The effects of the slip properties on green body

SiC ceramic slip should have the characteristics of high solid volume particles distribution, good stability and rheology. The solid volume fraction of the slip directly affects the strength and density of green body. Low viscosity is conducive to the elimination of bubbles in the slurry and the casting of complex parts. The slip's pH value, dispersant and solid phase volume fraction are closely related to the slip viscosity.

Adjusting the pH value in the slip can adjust the electric charge on the particles surface, so the electric charge increases, the electric double layer repulsion increases, and a stable state is achieved, shown as *Figure 10*.

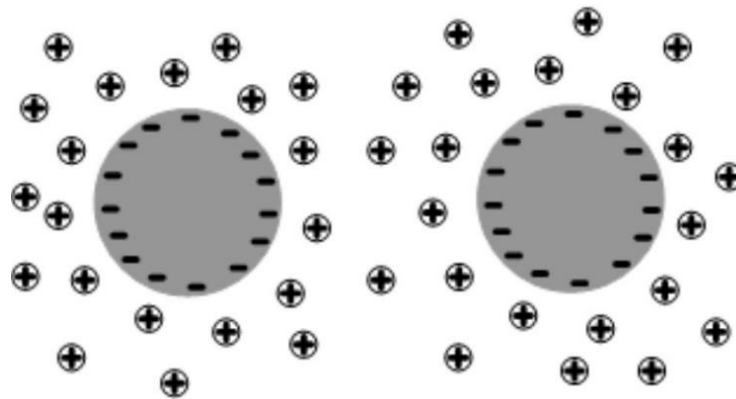
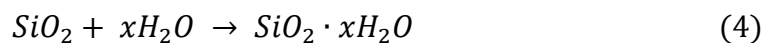


Figure 10 Electrostatic stabilization between two particles [25]

The chargeability of the surface of the SiC particle changes with the pH value of the slip. Sometimes, the electrostatic stability of the particles can be adjusted by adjusting the pH value without adding a dispersant [20].

The pH value has a great influence on the viscosity of the SiC slurry. The surface charge of SiC ceramic particles is different in different pH value aqueous media. This is because the surface of the SiC particles is generally coated with a layer of SiO_2 , and SiO_2 adsorbs water molecules in an acidic medium to form a hydrated layer $\text{SiO}_2 \cdot x\text{H}_2\text{O}$:



However, SiO_2 forms colorless SiO_3 in alkaline media, so that the surface of SiC particles has different properties in aqueous media with different pH values. The isoelectric point of SiC particles in slip is $\text{pH} = 3\sim 5$. In a large pH range, SiC particles are negative charge, and they have good electrostatic stability under alkaline conditions. Figure 2.13 shows the relation between the viscosity of SiC slip with solid-phase 32 vol% and pH value. The graph shows that the viscosity decline is relatively gently when NH_3 is added. When the pH is between

9.5~11.5, the viscosity will change quickly. When the pH is 11.5, the viscosity of the slip is lowest, which is about 72 mPa · s. Continue to add NH₃, the viscosity does not change much. Therefore, it is determined that pH=11.5 is the optimal pH value. The NH₃ increasing, makes the solutions negative ions, the surface negative charge of the SiC particles, and the double electric repulsion force increasing. So, this also increases the stability of the slip. When the amount of NH₃ is small, the negative ions in the solution does not increase much, and the repulsive force of electric double layer is not obvious, so the slurry viscosity decreases gently at the beginning. When the pH value is between 9.5~11.5, the negative ions in the solution increase rapidly, the surface potential of the SiC particles increases, and the repulsive force of the electric double layer increases rapidly, so the slurry viscosity decreases rapidly. Continue to add NH₃, the surface potential of SiC particles is almost unchanged, the repulsive force of the electric double layer no longer increases, almost unchanged slurry viscosity as a result. [20]

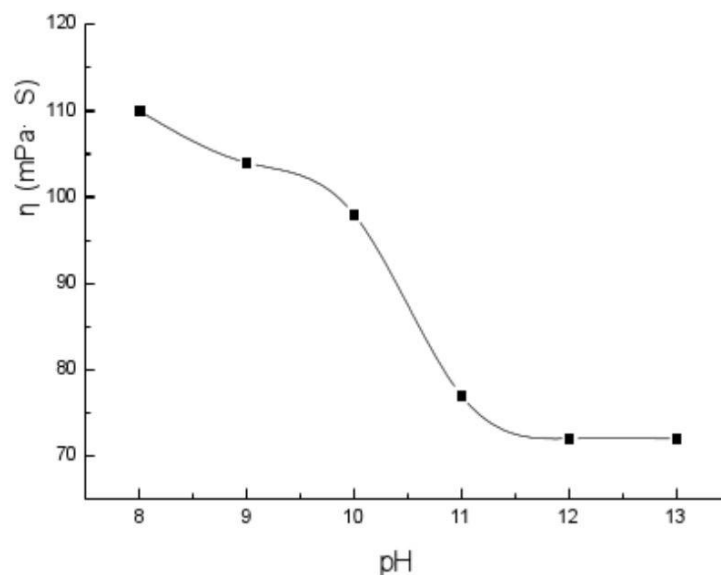


Figure 11 Viscosity & pH of 32vol% SiC slip with NH₃ [20]

2.5 The sintering process of silicon carbide ceramics

Sintering is the most important and unique process in the production of ceramic products. Ceramic materials are prepared from raw material powder, which has a high melting point and brittleness, so it can be prepared by sintering instead of high pressure or other processing. Sintering is a process to transform powder compacts into a strong final product by removing pores between particles. Through the entire sintering process can be divided into three different stages according to the changes in pore shapes and size.

During the sintering process, the shaped part shrinks and undergoes a decrease in porosity and an improvement in mechanical integrity. These changes are caused by the aggregation of powder particles into a denser substance during the sintering process. The sintering mechanism is shown in *Figure 12*. After pressing or casting, many powder particles come into contact with each other (*Figure 12a*). In the initial stage of sintering, necks are formed along the contact area between closed particles; In addition, grain boundaries are formed in each neck, and each gap between the particles becomes pores (*Figure 12b*). As the sintering progressing, the pores become smaller and more ball-shaped (*Figure 12c*). When the total particle area is reduced, the surface energy of the sintering driving force is larger than the grain boundary energy. Sintering is performed below the melting temperature, so there is usually no liquid phase. The mass transfer is achieved by atomic diffusion from the body particles to the neck region [26]. Sintering is a complicated phenomenon that occurs simultaneously in multiple processes, and it is also the simplest and best method to prepare ceramic materials.

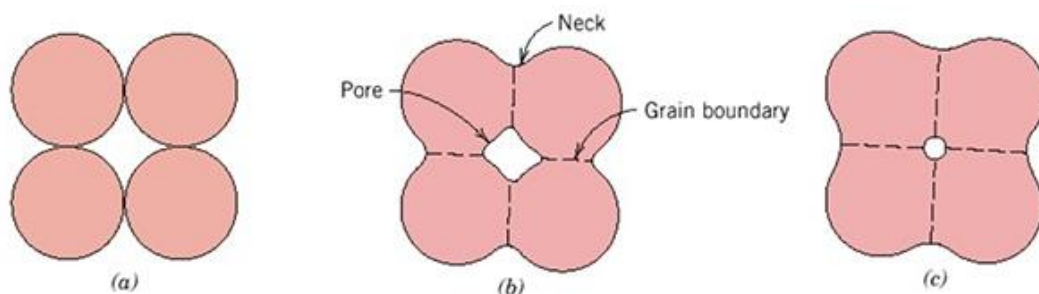


Figure 12 The changes of powder particles during the sintering process [26]

Ceramics are structural materials and the main reason is its heat resistance and wear resistance. In addition to the covalent bond materials like Si_3N_4 and SiC ,

few ceramics can maintain its strength without the usage of metal temperature [27].

The development of recrystallized silicon carbide (ReSiC or RSiC) is one of them. Recrystallized silicon carbide (ReSiC) is a kind of silicon carbide material without bonding phase, and its superior high-temperature performance is greatly related to its synthesis mechanism. The preparation method of the recrystallized silicon carbide material is as follows: using two kinds of high-purity silicon carbide with different particle sizes (coarse and fine powder) as raw materials, adding an appropriate amount of bonding agent (without adding sintering aids). After mixing according to a certain ratio, forming the mixture by slip casting or gel injection casting, under the high temperature which is 2200~2450°C, occurs evaporation-agglomeration recrystallization (*Figure 13*) between the silicon carbide particles [28]

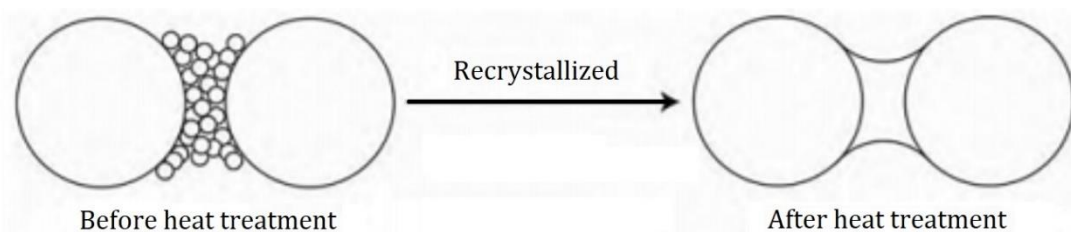
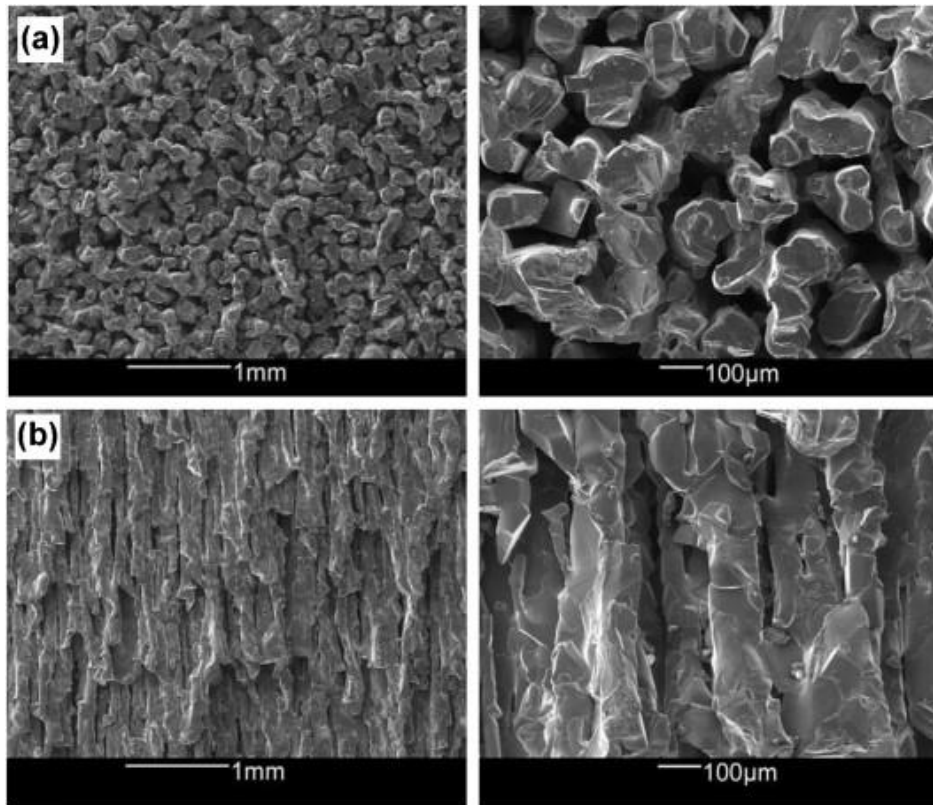


Figure 13: The recrystallization between SiC particles [28]

The particle symbiosis happened at the contact between the particles and formed a sintered body. Therefore, the recrystallized silicon carbide has no shrinkage and no liquid phase during the sintering process, and eventually forms a porous network structure with interconnected pores (*Figure 14*) [29].



*Figure 14: Microstructure of sintered SiC ceramic [30]
 (a) the radial section surface and (b) the axial section surface*

Since silicon carbide molecules can only produce a small amount of gas-phase molecules, it should be noted that the 'evaporation' here is not simply the vaporization of silicon carbide, but the decomposition of the silicon carbide body when the silicon dioxide (SiO_2) exist. When the fine particles occupy the gaps between the coarse particles, they will disintegrate during sintering, and then the large particles will agglomerate and deposit at the contact point to form a new grain boundary layer. So that the microstructure is strengthened, once the fine particles completely disappear and leave pores, the sintering process is completed [28].

2.6 Finishing

Some ceramic parts can be made into a finished product through the sintering process. However, it is more common that certain surfaces need to be machined to reach dimensional requirements, improve surface smoothness and eliminate surface defects. Machining accounts for a large portion of manufacturing costs, so it should be minimized and performed as efficiently as possible. Ceramic materials are difficult to process and expensive due to their high hardness and brittleness. Most ceramics cannot be successfully used for metal cutting tools because these tools are either not hard enough to cut ceramics or they break due to excessive local tensile loads. The tool must have a higher hardness than the ceramics, and must have a structure that can remove surface materials without causing excessive stress to the components [31].

3 Experimental

All SiC powders, dispersant, liquefying agents and equipment which were used in this experiment are provided by Washington Mills AS, Orkanger, Norway. The aims of this experiment are to measure the properties of SiC fine powder and its possible effects on SiC slip according to slip casting and sintering process for final SiC ceramic products, also to provide experimental data and reference for future research.

3.1 Silicon carbide powder used in the experiment

The detailed information of all SiC powders were provided by Washington Mills AS. The *Table 1* and *Table 2* shows the detailed particle size, with or without acid treatment, and their impurities. GW 100/F (*Figure 15a*) are coarse powder which also can see with its particle size. The rest of the powders are FPG 7 (Fine powder Green with surface area $\sim 7 \text{ m}^2/\text{g}$) as shown in *Figure 15b*.

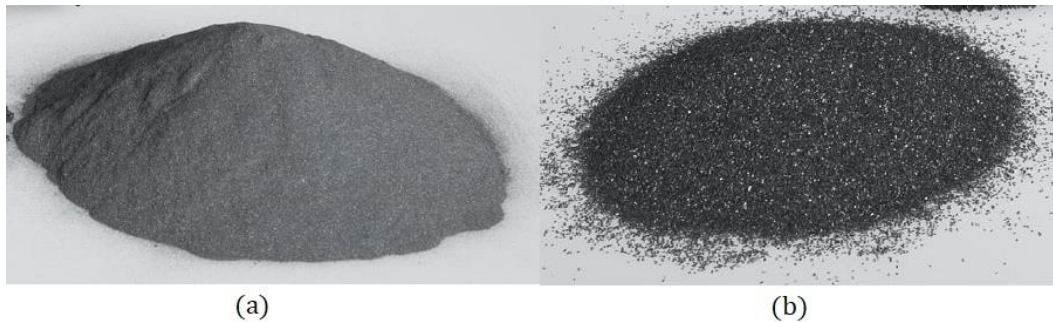


Figure 15: SiC fine and coarse powders. (a) FPG 7 and (b) GW 100/F.

Table 1: Particles sizes and acid-treated of SiC powders with their lot numbers.

	Lot nr.	SiC particle size (μm)				Acid treated
		d_0	d_{10}	d_{50}	d_{90}	
FPG 7	139348	16.41	8.37	2.46	0.38	Yes
	142939	16.28	7.95	2.59	0.39	Yes
	140416	16.40	8.15	2.76	0.42	Yes
	142851	14.02	7.37	2.58	0.40	Yes
	142985	14.45	8.02	2.67	0.41	Yes
	62330	16.36	8.17	2.61	0.41	No
	62327	16.34	7.99	2.54	0.42	No
	GW 100/F	48.35	54.90	100.47	159.82	No

Fine powder particle size distribution measured on Malver Mastersizer.'

GW 100/F particle size distribution measured on RoTap.

Table 2: Impurities content of SiC powders.

Lot nr.	Impurities (%)							
	Al	Ca	Fe	Free C	Free Si	O	SiC	SiO ₂
139348	0.017	0.001	0.077	0.460	0.160	0.830	98.2	1.090
142939	0.013	0.001	0.090	0.520	0.066	0.940	98.1	1.240
140416	0.022	0.003	0.201	0.460	0.094	1.010	98.0	1.250
FPG 7 142851	0.017	0.001	0.110	0.540	0.091	0.960	98.2	1.070
142985	0.011	0.001	0.080	0.510	0.075	0.930	98.0	1.330
62330			0.704					
62327			0.715					
GW 100/F	0.003	0.003	0.020	0.07	0.05		99.5	0.20

Free C, Free Si, SiO₂ measured according to FEPA-Standard 45-1: 2011.

Fe, Al, Ca measured with XRF.

Oxygen measured by combustion and IR detection.

3.2 Conductivity measurement

The FPG 7 were used for this conductivity measurement with lot numbers 139348, 142939 and 140416.

The slip was prepared by pouring the 5 grams FPG7 and 100 ml deionized water into a 200 ml beaker, and stirring it with a plastic spoon for 2 minutes. Stirring with a plastic spoon, because the metal spoon could affect the conductivity value. The well-mixed slip was measured at room temperature using a conductivity meter (*Figure 16*) with a measuring cell. The measuring cell (*Figure 16*) was immersed ca. 7 cm in the slip. The slip temperature and conductivity were analyzed by conductivity meter, and the values were shown on the screen. The stable values were recorded when the display indicator was no longer flashes. The procedure above was repeated to measure each FPG7 powder with different lot number.



Figure 16: WTW Inolab Cond. 7110 conductivity meter (left) and its measuring cell (right) [32].

3.3 pH measurement

The pH value was determined by five FPG7 powders with lot numbers were 142851, 142985, 142939, 62330 and 62327.

The slips of pH measurement were prepared by adding 10 grams powder to 90 ml deionized water in a 100 ml beaker. The slip was stirred by a rod stirrer (*Figure 17 b*) with speed 9 for 30 seconds, and then reduced speed to 1. The pH electrode (*Figure 17 c*) was immersed in the slip, and the actual pH of the slip from the display were read off. The value was stable when the arrow no longer blinks. The five FPG7 powders were measured one by one with the same method above. The pH results were exported to Excel.

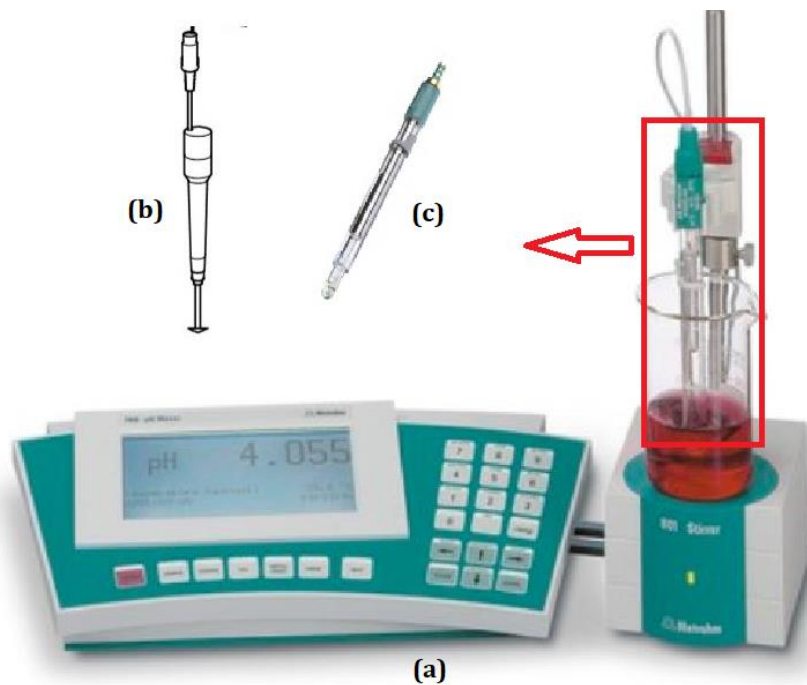


Figure 17: (a) 780 pH meter, (b) rot stirrer, and (c) pH electrode [33].

3.4 Viscosity measurement

The two powders were used in this measurement are GW 100/F and FPG7 with lot number 139587. The AMP-dispersant used for stabilization of slip in this work. The chemical name of AMP-dispersant is 2-amino-2-methyl-1-propanol (*Table 3*). It is a colourless liquid which is used to additives for paints, pigment dispersion and pH adjustment. AMP-dispersant is miscible with water and soluble in alcohol. AMP- dispersant is also provided by Washington Mills AS.

Table 3: Characteristics of AMP-dispersant [34].

Chemical formula	C ₄ H ₁₁ NO
Molecular Weight	89.14 g/mol
Specific gravity	0.94
Density	0.934 g/ml at 25 °C
pH (5.1% solution)	9~10.5
Physical appearance	Liquid
Boiling point	ca 165 °C

Four 500 ml test bottles (*Figure 19*) were prepared and marked with A, B, C and D on the outside of the bottle. Poured 50 ml of deionized water into bottle A and bottle B, and added one drop of dispersant to bottle A and four drops of dispersant to bottle B. Poured 30 ml of deionized water into bottle C and bottle D, and added one drop of dispersant to bottle C and four drops of dispersant to bottle D. The four solutions were hand stirred. Immediately after this, 100 grams of FPG7 and 100 grams of GW 100/F were poured into each bottle. Stirred the slips vigorously to make it well-mixed. The slip was ca. 9 cm high from bottle lowest (*Figure 19*). The proportion of coarse and fine powder was 50%-50%. The slip A and B were 80wt% of total solid content, and the slip C and D were 87wt%. Since AMP dispersant were liquid, so the weight was measured with its drops amount. A drop of AMP-dispersant was 0.03 gram. *Table 4* and *Table 5* described the composition of four slips and the amounts of liquefying agents in the slips.

Table 4: The composition of the slip samples.

Sample No.	A	B	C	D
GW 100/F	100 g	100 g	100 g	100 g
FPG 7	100 g	100g	100 g	100 g
water	50 ml	50ml	30 ml	30 ml
AMP	0.03 g	0.12 g	0.03 g	0.12 g

Table 5: The amounts of the liquefying agents in the samples.

Sample No.	A	B	C	D
water	20 %	20 %	13 %	13 %
AMP	0.012 %	0.048 %	0.012 %	0.048 %

Slip viscosity was measured with a rotating spindle viscometer (Brookfield DV-II+, USA). The Brookfield viscometer were provided with a set of four spindles (*Figure 18*) with two cylindrical spindles (nr. 61, 64) and two-disc spindles (nr. 62, 63). The spindle were inserted and centered in the slip until the slip's level is at the immersion groove (the red circle mark on *Figure 18*) in the spindle's shaft. The depth of the spindle in the slip was also different, the spindle depth in the slip were shown in *Figure 18* (marked in black). Spindles were used one by one in each slip with the constant rate 100 RPM and was rotated for one hour. The values were recorded every five minutes.

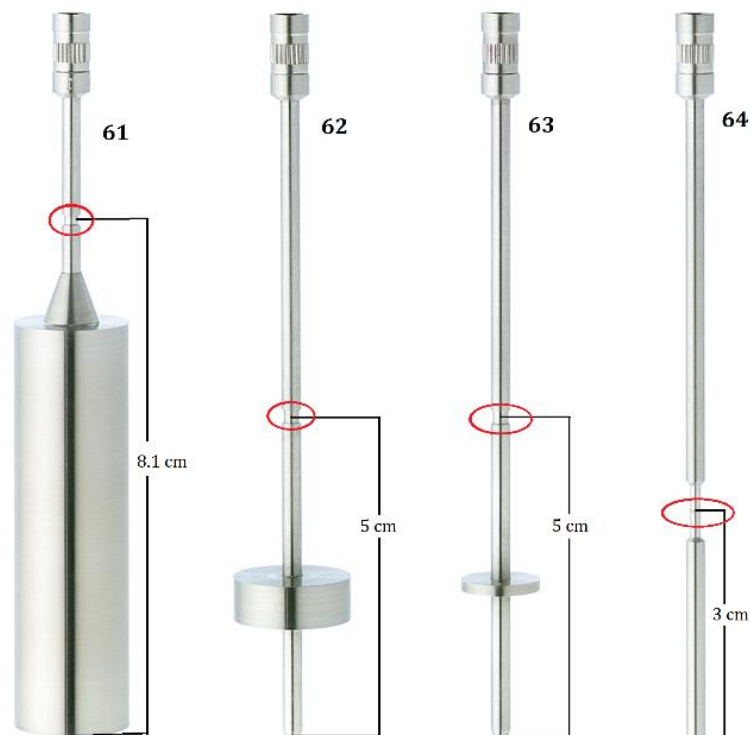


Figure 18: The four standard spindles of Brookfield DV viscometer, marked the immersion grooves and depth in the slip



Figure 19: Test bottle with its height ca. 10 cm, and the slip height ca. 9 cm.

4 Results

After performed measurements, the conductivity, pH and viscosity values were uploaded to Excel, where the complete data and results are ready for analysis.

4.1 Conductivity of FPG 7 powder

The conductivity results of FPG7 are shown in *Table 6* and the column chart *Figure 20*. The final conductivity was determined by slip conductivity minus the conductivity of deionized water. The average value of FPG7 conductivity is ca. 13.0 $\mu\text{S}/\text{cm}$.

Table 6: The conductivity results of FPG7.

Lot nr.	142939	140416	139348
Temperature ($^{\circ}\text{C}$)	22	22	22
Slip conductivity ($\mu\text{S}/\text{cm}$)	16.0	9.9	14.6
Deionized water conductivity ($\mu\text{S}/\text{cm}$)	0.6	0.6	0.6
Final conductivity ($\mu\text{S}/\text{cm}$)	15.4	9.3	14.0

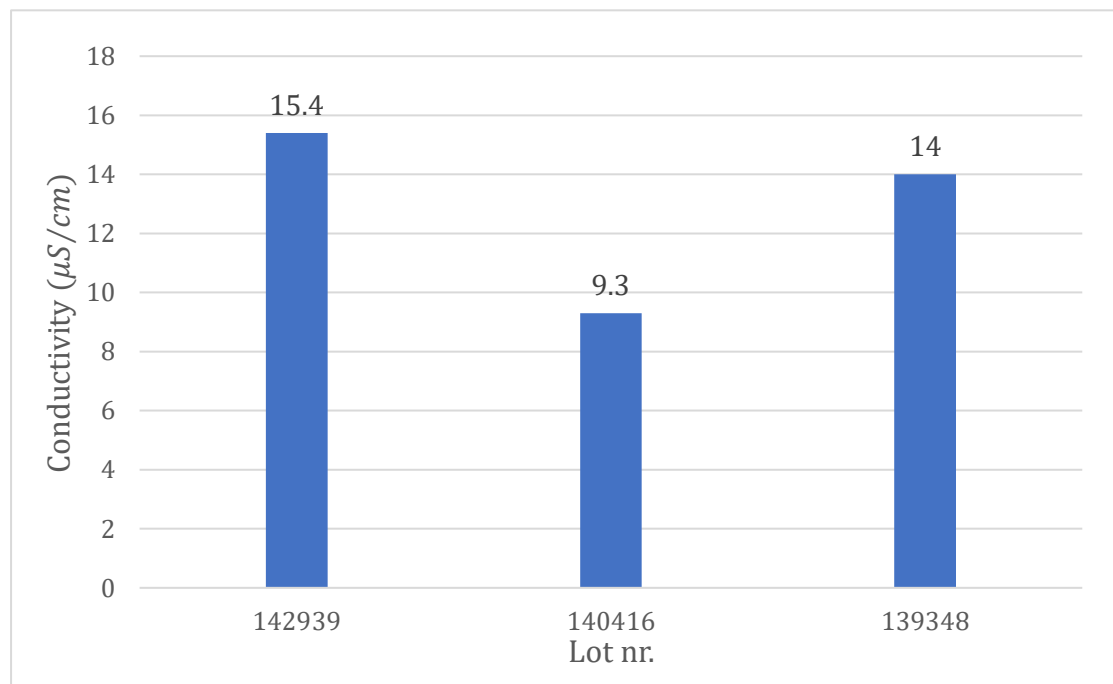


Figure 20: The conductivity of three FPG 7 powders.

4.2 pH of FPG7 powder

The results of pH measurement are shown in *Table 7* and *Figure 21*. The 142851, 142985 and 142939 has lower pH, average pH = 4.30. The 62330 and 62327 has higher pH, average pH = 6.41.

Table 7: The pH value of five FPG 7 powders.

Lot nr.	142851	142985	142939	62330	62327
pH	4.29	4.34	4.26	6.43	6.38

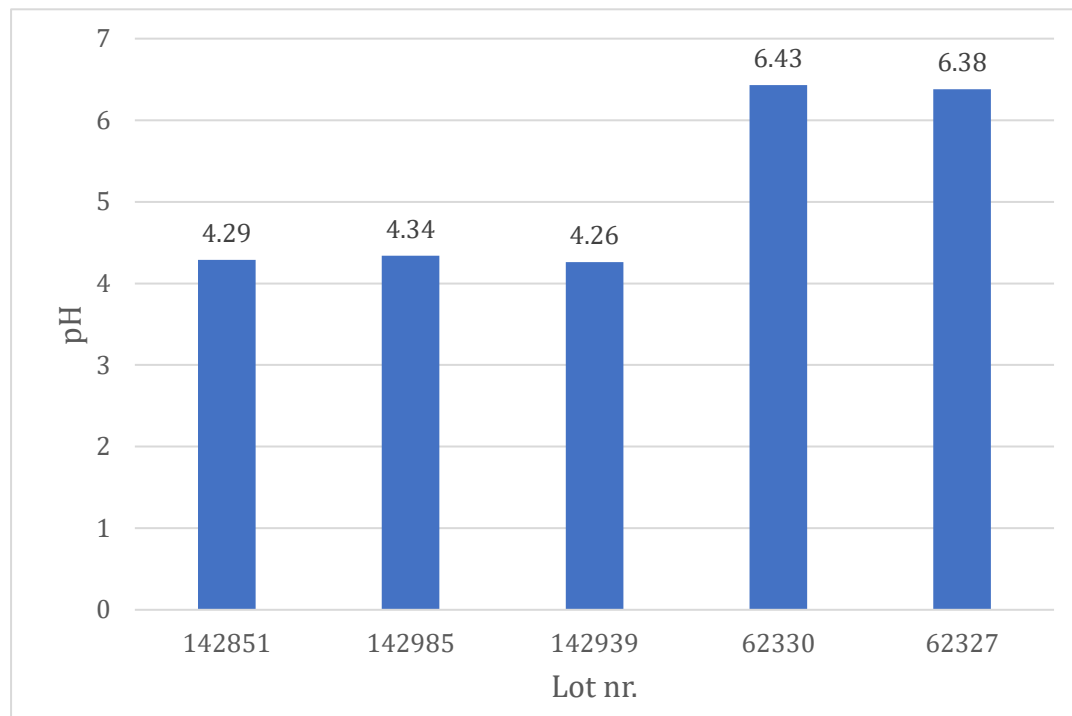


Figure 21: The pH value of five FPG 7 powders.

4.3 Experimental results and laboratory data of FPG 7 powder

Due to the impact of the Covid-19 epidemic, the laboratory was closed and continue measurement was not possible. Fortunately, Washington Mills AS provided more laboratory data of SiC powder. The results are shown in *Table 8* and *Figure 22*. As shown in the figure, the fine powder with lot nr 62330 and 62327 had higher conductivity and pH value. The rest of the powder had correspondingly lower values.

Table 8: Summary of experiment results and provided results.

Lot nr.	139348	142939	140416	142851	142985	62330	62327
Cond.($\mu\text{S}/\text{cm}$)	14	15.4	9.3	13.6*	17.0*	40.1*	40.9*
pH	4.48*	4.26*	4.82*	4.29	4.34	6.43	6.38

*Results provided by Washington Mills AS

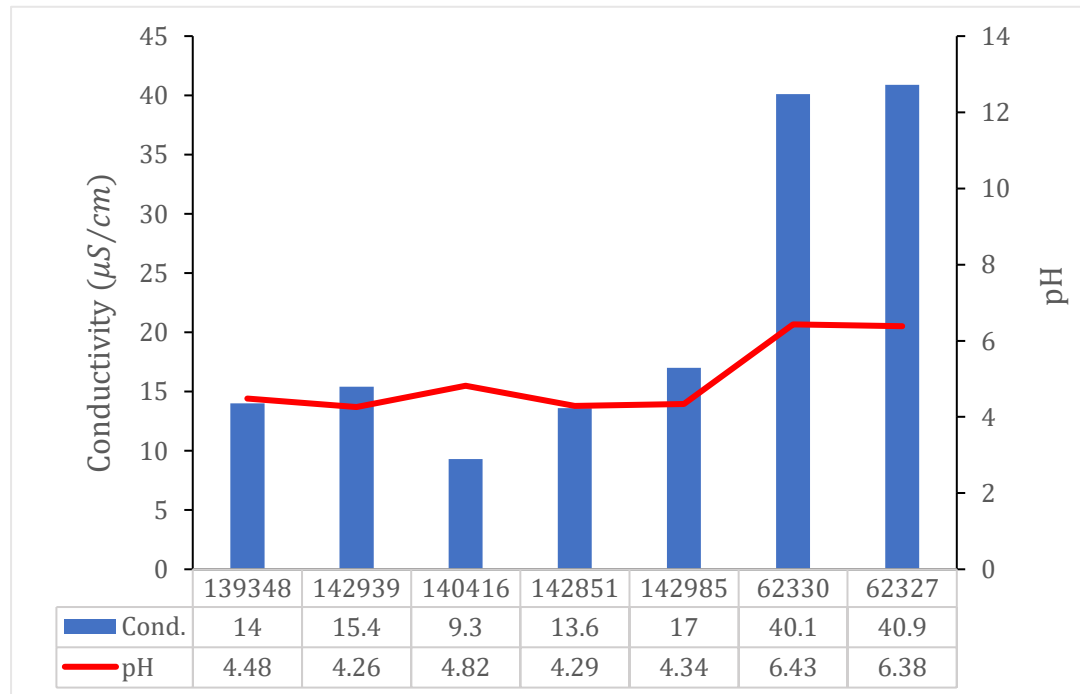


Figure 22: Histogram of conductivity & pH.

4.4 Viscosity of SiC slip

After the viscosity experiment was completed, the experimental results with different spindle operations were obtained.

The viscosity results of slip A with four spindles are shown in *Table 9, Figure 23*. Slip A contains 80 wt% of solid phase, 20 wt% water and 0.012 wt% AMP-dispersant. The viscosity in spindle 61 was over the measuring range and the spindle 64 was lesser than 10 % torque, which had no result. As shown in *Figure 23* that the viscosity of spindle 62 is lower than spindle 63.

Table 9: Viscosity of slip A.

Time (min)	0	5	10	15	20	25	30	35	40	45	50	55	60
61 Viscosity (cP)	Over range												
62 Viscosity(cP)	39	44	72	95	106	112	122	135	161	183	210	222	262
63 Viscosity(cP)	<10% torque		161	175	191	203	215	224	255	266	266	274	281
64 Viscosity(cP)	< 10% torque												

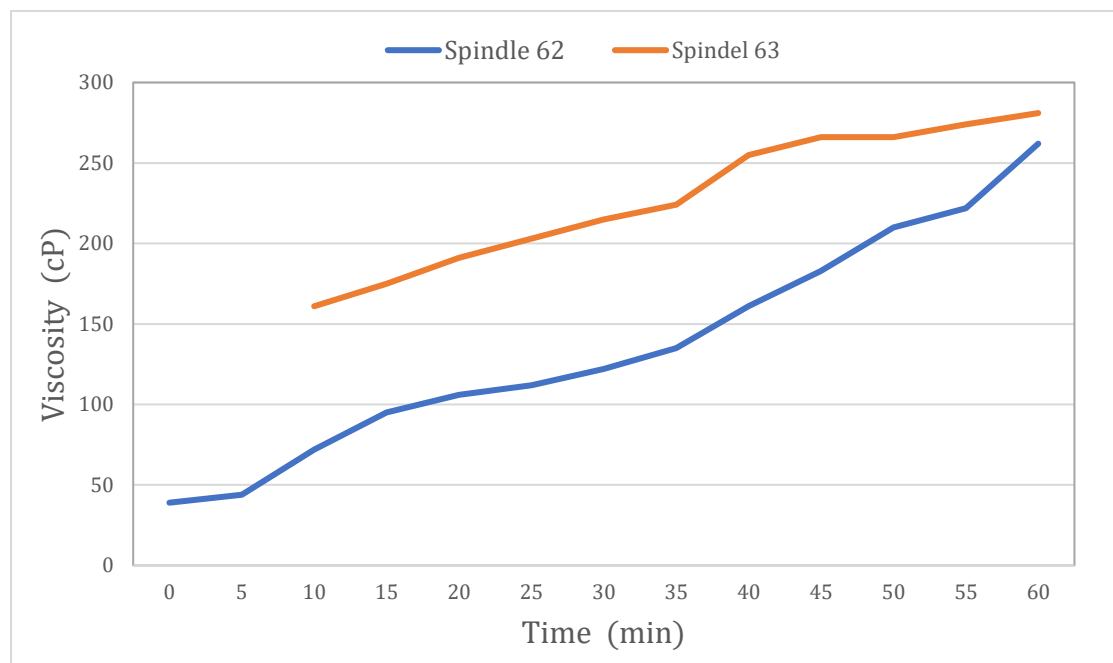


Figure 23: Viscosity of slip A.

The viscosity results of slip B with four spindles are shown in *Table 10, Figure 24*. Slip B contains the same solid phase and water, but contains 0.048 wt% AMP-dispersant which are three times more than slip A. The viscosity in spindle 61 was over the measuring range, and the spindle 62 was also over range after 30 minutes. As shown in *Figure 24* that the viscosity of spindle 62 is lower than spindle 63, and spindle 64 has the highest viscosity.

Table 10: Viscosity of slip B.

Time (min)	0	5	10	15	20	25	30	35	40	45	50	55	60
61 Viscosity (cP)	Over Range												
62 Viscosity(cP)	34	135	190	208	214	215	235	Over range					
63 Viscosity(cP)	207	280	310	320	330	340	344	366	356	360	356	369	365
64 Viscosity(cP)	695	980	1080	1160	1236	1230	1240	1260	1150	1146	1260	1300	1326

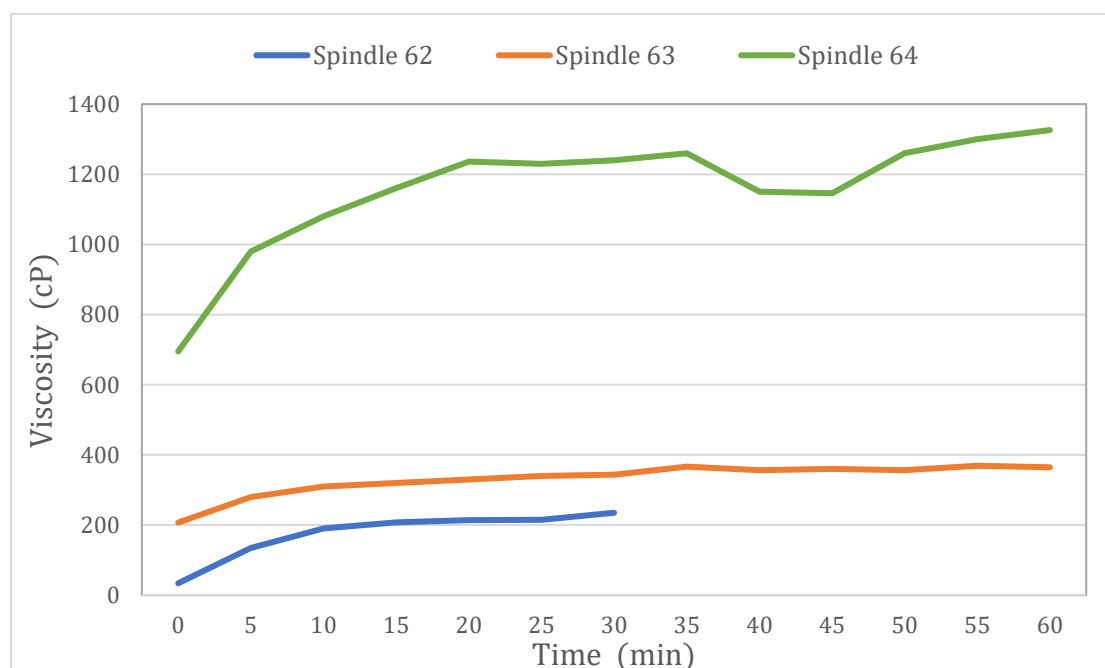


Figure 24: Viscosity of slip B.

The viscosity results of slip C with four spindles are shown in *Table 11, Figure 25*. Slip C contains 87 wt% of solid phase, 13 wt% water and 0.012 wt% AMP-dispersant. The viscosity in spindle 61 was over the measuring range, spindle 62 was also over range after 10 minutes, and the spindle 64 was lesser than 10 % torque, which didn't have any result. As shown in *Figure 25* that the viscosity of spindle 63 is lower than spindle 62 in the first 10 minutes.

Table 11: Viscosity of slip C.

Time (min)	0	5	10	15	20	25	30	35	40	45	50	55	60
61 Viscosity (cP)	Over range												
62 Viscosity(cP)	242	244	282	Over range									
63 Viscosity(cP)	219	210	217	230	250	277	311	349	389	450	538	722	680
64 Viscosity(cP)	< 10% torque												

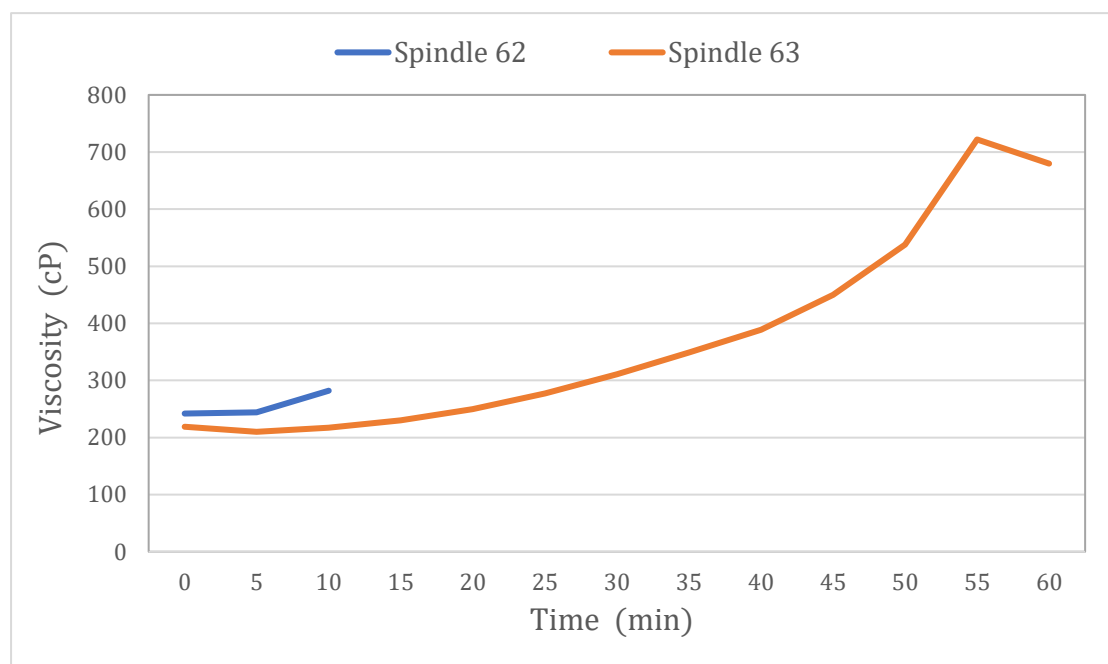


Figure 25: Viscosity of slip C.

The viscosity of slip D with four spindles are shown in *Table 12, Figure 26*. Slip D contains the same solid phase and water with slip C, but contains 0.048 wt% AMP-dispersant which are three times more than slip C. The viscosity in spindle 61 was over the measuring range, the spindle 62 was also over range after 10 minutes, and the spindle 64 was less than 10% torque. The *Figure 26* shows that the viscosity of spindle 63 is lower than spindle 62 in the first 10 minutes.

Table 12: Viscosity of slip D.

Time (min)	0	5	10	15	20	25	30	35	40	45	50	55	60
61 Viscosity (cP)	Over range												
62 Viscosity(cP)	198	204	241	Over range									
63 Viscosity(cP)	182	172	179	187	202	222	247	278	323	361	426	508	623
64 Viscosity(cP)	< 10% torque												

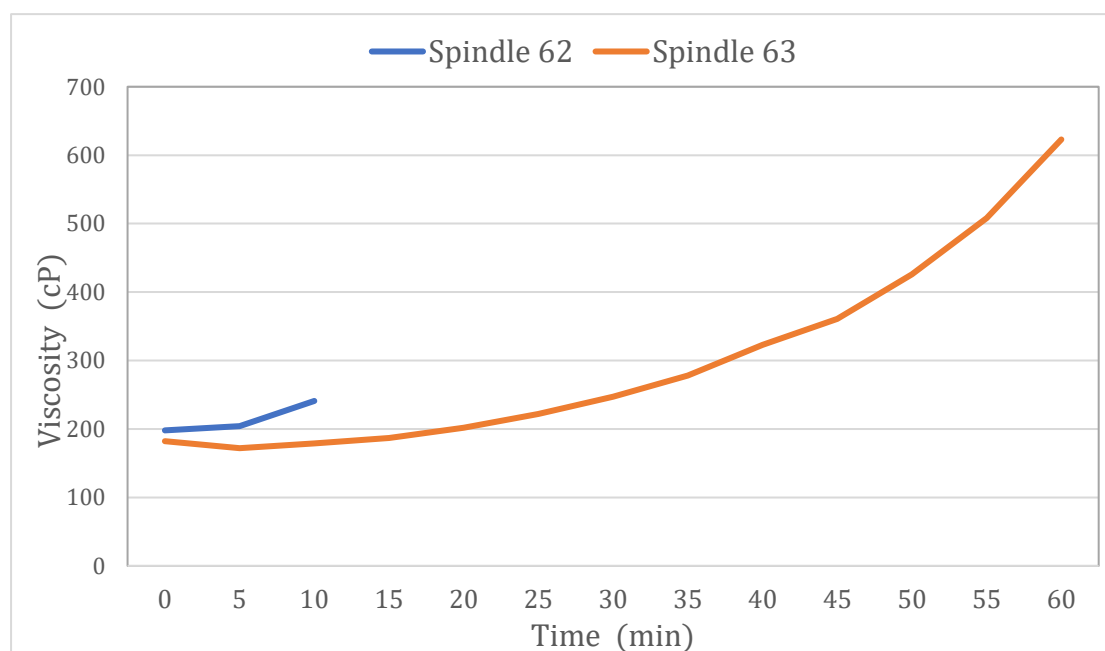


Figure 26: Viscosity of slip D.

Since spindle 62 and 63 had better results. So the SiC solid content and AMP-dispersant amount on the viscosity were plotted as *Figure 27* and *Figure 28*. The *Figure 27* shows the slip A and B with 80 wt% solid content, had lower viscosity than slip C and D, especially slip A with 0.03 g AMP-dispersant. For the spindle 63, the viscosity of slip A and B are equable, slip C and D are increased by time. Still, slip A has the lowest viscosity of all slips as shown in *Figure 28*.

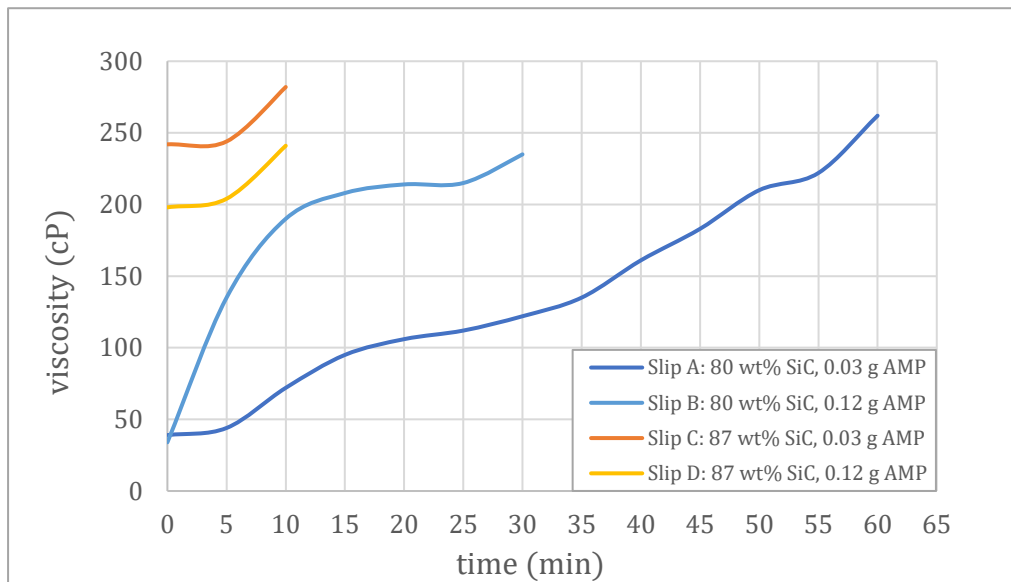


Figure 27: SiC wt% and dispersant amount on the viscosity of the spindle 62 under various time.

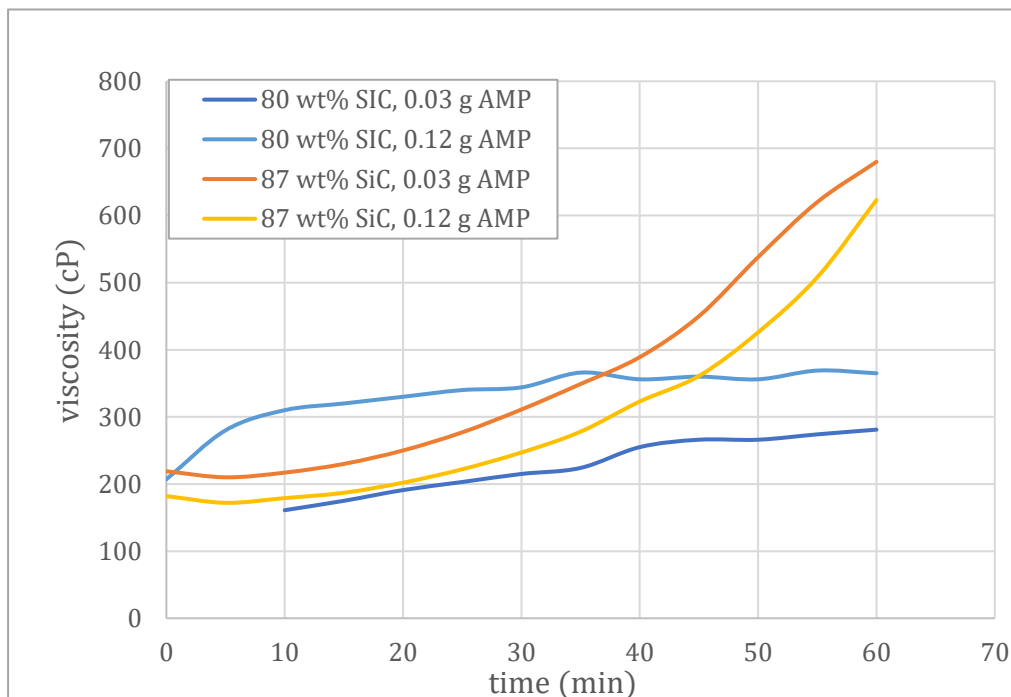


Figure 28: SiC wt% and dispersant amount on the viscosity of the spindle 63 under various time.

5 Discussion

5.1 Effect of conductivity and pH on FPG 7 powder

The SiC fine powder contains impurities such as aluminum, calcium, iron, free carbon, free silicon and oxygen (*Table 2*). When the powder was combined with deionized water, the irons and salts from the particle surface was dissolved in the water. The *Figure 22* shows the conductivity with the last two slips (62330 and 62327) was higher than the first five slips (139348, 142939, 140416, 142851 and 142985). The reason is the iron contents of the last two slips were higher. This also shows in *Table 2* that the last two powders, 62330 and 72327, had higher iron content than the rest of the powders. The higher conductivity measure, the more irons have been dissolved from powder into the water. At the same time, the pH of the last two powders was also higher than the other powders, the average pH is 6.41, and the other powders pH is 4.44. The reason is the acid washing process of the powder. The pH of the slips with acid-washed powders are reduced, while the pH of the slips with unacid-washed powders are relatively high.

5.2 Effect of viscosity on SiC slip

During the viscosity measurement, observed that the slip was precipitated over time. The longer the time, the more obvious the precipitation phenomenon, and the thicker the precipitation layer at the bottom of the slip. Slip A and B measurements were first completed, slip C and D were measured one week after. Observed that the slip A and B were precipitated, after the hard hand stirring, the original slip state was restored. But the sediment layer of slip C and D, were solidified into a bard block with ca. 1 cm thick at the bottom of the bottle. So, the slip C and D were re-prepared and then measured for viscosity. The precipitation phenomenon in the slip could lead to the upper layer of slip got thinner over time, and if the depth of spindle also in the upper layer, so a decreased viscosity as a result. Similarly, the bottom layer of the slip became more and more viscous by particle settling, and the spindle depth is in the slip's lower layer, which also caused the viscosity increased or over the test range. It is can see from *Figure 27* and *Figure 28* that the stability of the slip A and B has further improved with the 80 wt% solid content, there is not much change in the viscosity value over the entire range of dispersant from 0.03-0.12 g because of the flocculated layer formed during the time.

It also can see *Figure 27* and *Figure 28* that the slip A and B with the same 80 wt% solid content, the stability of the slip with 0.03 g of dispersant was the best. But in the same 87wt% solid content, slip C and D, with 0.12g of dispersant had the best stability.

5.3 Recommendations for further work

The combination of theory and experiment which have been down by preparing and measuring SiC slip and obtained some measurement data can be used as a reference for subsequent research. Due to the impact of the Covid-19 epidemic, the laboratory was closed. The conductivity and pH of the silicon carbide slip in the viscosity experiment could not be tested.

5.3.1 The measurement of slip stability

It is recommended to prepare SiC slips with the same ratio of solid phase with a wetting agent to complete the conductivity and pH test. Also, can re-measure the viscosity of the slip, analyze the conductivity and pH to obtain the best slip with high solid phase, well-dispersed and high stability. Besides the viscosity measurement, pay extra attention to sedimentation and solidification of the slip over time. Such as the sedimentation height of the upper liquid and the lower sedimented particles, the duration of the sedimentation phenomenon stops, and the thickness of the solidification. Observation by sedimentation indicates the stability of the SiC powder slip under the conditions of different pH values and different solid-phase contents. As a suggestion, the well-dispersed SiC slip places in a measuring cylinder, let it stand for a while, observe the height of the upper clear liquid. The height of the clear liquid indicates the stability of the slip. The greater the height, the greater the powder settling height in the slip, and the more unstable the slip.

5.3.2 Viscosity measurement and rheology of SiC slip

It is recommended to carefully look up the information about non-Newtonian fluids and the relationship between shear rate, shear stress, torque and viscosity before the viscosity test. Through plotting the shear stress as a function of shear rate from the viscosity test, the fluid type can be determined. And also, the fluid properties and fluid stability of SiC slip can be distinguished. It is convenient to prepare slips for improving stability and reducing settlement. A data (from Institute of Ceramics and Powder Metallurgy, Northeastern University [4]) indicated that SiC slip is Bingham fluid, which belongs to the non-Newtonian plastic fluid. Since the experiments in this paper have no relevant results on

shear rate, shear stress and torque, it is impossible to conclude that SiC slip is Bingham fluid. A guidebook from Brookfield called *More Solutions to Sticky Problems* could give more advice about the viscosity measurement with different type of fluid.

With viscosity measurement, recommended to use disc-spindles which are spindle 62 and 63 from spindle set of Brookfield DV-II+ viscometer, because the measurement with these two spindles shows the better results. The cylindrical spindles such as spindle 61 and 64, are not recommended to use on the measurement because spindle 61 were over test range and spindle 64 had less than 10% torque.

5.3.3 Complete SiC slip casting process

After prepared slips with different amount of dispersant and solid content, use plaster to make several molds which have the same form, and perform slip casting. The green bodies remove after slip casting and observe the difference in the internal structure with a microscope. The performance of the slip, the properties of the plaster mold and the operation procedures will affect the quality of the finished product of slip casting. Through research and observation of the green body, the shortage of the slip preparation can also be analyzed. For example, the cracking of the green body is due to the poor quality of the slip, the ageing time is not enough, or the green body stayed too long in the plaster mold. Poor formation of the green body is caused by insufficient electrolyte in the slip, or excessive electrolyte lead to agglomeration. The difficulty of mold unloading is because the slip liquid content is too high, or the powder content is too much. And the bubbles and pinholes in the slip caused by the high viscosity that the air bubbles not being discharge [35]. By studying the green body's defects and the slip's properties, can help to prepare better SiC slip to produce the qualified SiC products.

5.3.4 Selection and dosage of dispersants

The dispersant type affects the viscosity of the SiC slips. The dispersant such as acacia gum, PEG (polyglycol), TMAH (tetramethyl ammonium hydroxide), water glass (sodium silicate), PMAA-NH₄ (poly (methacrylic acid)), STPP (sodium tripolyphosphate). A study (from Northeastern University, China. [20]) shows that these dispersants can disperse SiC slip to different degrees. The method was to mix SiC powder with deionized water to configure a slip with a solid content of 32 vol% then added a certain amount of dispersant. Analyzed the relation between the viscosity change and dispersant's amount for determine the optimal addition amount. Compared different curves corresponding to different dispersants to determine the best dispersant for the slip. The experimental

results showed that TMAH and PMAA have the greatest influence on the viscosity of SiC slip. When the amount of TMAH is 2 wt% were added, the slip with 32 vol% had the best fluidity, and the viscosity decreases from 110 mPa · s to 38 mPa · s. [20]. Also, in another study (from Harbin University of Science and Technology, China), it was pointed out that PMAA- NH_4 has better dispersibility in aqueous slip. the best addition amount was 0.2-0.3 wt% with pH 9-10. A sterically hindered dispersant PEI makes the particles generate repulsive force to achieve the dispersion effect. Added 1 wt% PEI at pH 10.38 had the best dispersion effect, and best fluidity with a 30-60 vol% ethanol solution. In the end of the study, it was studied SDS-dispersant (sodium dodecyl sulfate) and CTAB-dispersant (Hexadecyl trimethyl ammonium bromide) as well. It was pointed out that 0.01g/L SDS and 0.10g/L CTAB in the SiC slip made SiC particles stably dispersed and suspended. But there are few research on SDS and CTAB dispersant in SiC slip [36].

Therefore, it is recommended to use TMAH and PMAA as dispersants. Disperse ca. 5 wt% TMAH or PMAA in the slip with 80 wt% solid content to measure the viscosity. Then compare the data to see if it has a great effect on viscosity.

6 Conclusions

The aim of this bachelor project was to study how surface properties such as conductivity and pH will affect slip properties of SiC production, and to measure conductivity, pH and viscosity of SiC slip with different amount of dispersant and solid content. In order to improve SiC slip with high stability for slip casting process. The measurement was performed in the laboratory of Washington Mills AS.

The result and discussion in this thesis enable the following conclusions to be drawn:

(1) The acid washing process of SiC fine powder has a significant effect on the conductivity and pH of SiC slip. The SiC slip with unacid-treated powder has high conductivity because of higher iron content. The SiC slip with acid-treated powder has relatively lower conductivity. The conductivity was decreased from 40.5 $\mu\text{S}/\text{cm}$ to 13.9 $\mu\text{S}/\text{cm}$ and pH from 6.41 to 4.44.

(2) When AMP (2-amino-2-methyl-1-propanol) as the dispersant, the slip with 80 wt% solid content with 0.012 wt% AMP-dispersant had the best slip stability.

(3) The viscosity measured by disc-spindle nr 62 and 63 (from Brookfield DV-II+ viscometer) increases over time because of the sedimentation of slip particles.

There are some recommendations for further research:

(1) Re-measure the viscosity test and observe the settlement and solidification in the slip.

(2) Study the rheological properties of SiC slip and confirm whether the fluid is Bingham plastic fluid or other types of fluid.

(3) Discover the new method to measure viscosity and recommend using disc-spindles.

(4) Perform slip casting process on SiC slip and study the slip effect on the green bodies.

(5) Use different dispersants with different amount to prepare SiC slip with good stability and high solid content.

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Appendix

Appendix A: Risk Assessment from NTNU

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 NTNU HMS	Kartlegging av risikofyllt aktivitet	Utarbeidet av	Nummer	Dato	
		HMS-avd.	HMSRV2601	3/22/2011	
		Godkjent av	Side	Erstatter	
		Rektor		12/1/2006	

Enhet: Institutt for materialteknologi **Dato:** 10.02.2020
Linjeleder: Tor Grande
Deltakere ved kartleggingen (m/ funksjon): Kjersti Kleveland, Anne Marie M. Moe, Bjørn almi, Rongling Yuan
 (Ansv. veileder, student, evt. medveiledere, evt. andre m. kompetanse)
Kort beskrivelse av hovedaktivitet/hovedprosess: Bacheloroppgave student Rongling Yuan. Avanserte keramer for fremtiden.
Er oppgaven er rent teoretisk? (JA/NEI) NEI
 JA betyr at veileder inntar for at **oppgaven ikke inneholder noen aktiviteter som krever risikovurdering**. I dette tilfellet er det ikke nødvendig å fylle ut resten av skjemaet
Skal du motta prøver fra industri? (JA/NEI) JA
 JA betyr separat risikovurdering av prøvene individuelt
Er det trygt å utføre arbeidet utenfor normal arbeidstid (8-17)? (JA/NEI) JA
Signaturer: *Ansvarlig veileder:* Kjersti Kleveland *Student:* Rongling Yuan

ID nr.	Aktivitet/prosess	Ansvarlig	Eksisterende dokumentasjon	Eksisterende sikringstiltak	Lov, forskrift o.l.	Kommentar
1	Konduktivtets måling	Rongling Yuan		Støvmaske, engangshansker og vernebriller	n/a	Engangshansker, lab frakk, støvmaske, vernebriller og innesko er påkrevd
2	pH-måling	Rongling Yuan		Støvmaske, engangshansker og vernebriller	n/a	Engangshansker, lab frakk, støvmaske, vernebriller og innesko er påkrevd.
3	Viskositetsmåling	Rongling Yuan		Støvmaske, engangshansker og vernebriller	n/a	Engangshansker, støvmaske, vernebriller, innesko og lab frakk er påkrevd.
4	Forberede gipsformer	Rongling Yuan		Engangshansker og vernebriller	n/a	Engangshansker, støvmaske, vernebriller, innesko og avtrekshetter er påkrevd.
5	Slip Casting	Rongling Yuan		Hansker, vernebriller	n/a	Hansker og vernebriller er påkrevd.

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2/21/2020 Side 2 av 6

 NTNU HMS /KS	Risikovurdering	Utarbeidet av	Nummer	Dato	
		HMS-avd.	HMSRV2603	2/4/2011	
		Godkjent av	Side	Erstatter	
		Rektor		2/9/2010	

Enhet: Institutt for materialteknologi **Dato:** 10.02.2020
Linjeleder: Tor Grande
Deltakere ved risikovurderingen (m/ funksjon): Kjersti Kleveland, Anne Marie M. Moe, Rongling Yuan
 (Ansv. veileder, student, evt. medveiledere, evt. andre m. kompetanse)
Risikovurderingen gjelder hovedaktivitet: Bacheloroppgave student Rongling Yuan. Avanserte keramer for fremtiden.
Signaturer: *Ansvarlig veileder:* Kjersti Kleveland *Student:* Rongling Yuan

ID nr.	Aktivitet/prosess fra kartleggingskjemaet	Mulig uønsket hendelse	Vurdering av sannsynlighet (1-5)	Vurdering av konsekvens				Risiko-verdi (menneske)	Kommentarer/ status Forslag til tiltak
				Menneske (A-E)	Ytre miljø (A-E)	Øk./ materiell (A-E)	Om-dømme (A-E)		
1	Konduktivtets måling	Puste de løse SIC pulver	1	A	A	A	A	A1	Støvmaske og vernebriller må brukes for å forhindre at pulver kommer inn i nesen og øyene.
2	pH-måling	Puste de løse SIC pulver	1	A	A	A	A	A1	Støvmaske og vernebriller må brukes for å forhindre at pulver kommer inn i nesen og øyene.
		Hudirritasjon pga berøring av bufferløsning	1	A	A	A	A	A1	Bruk vernebrille med sidevern, egnede varnehansker. Legg inn rekreasjonsfaser til regenerasjon av huden. Bruk av forebyggende hudbeskyttelse (hudkrem/salver) anbefales.

3	Viskositetsmåling	Puste de løse SIC pulver og hudirritasjon pga berøring av dispergeringsmidler	1	A	A	A	A	A1	Bruk vernebrille med sidevern. Bruk egnede vernehansker. Kjemikaliehansker testet i henhold til EU 374 er egnet, også pustemaske er nødvendig.
4	Forberede gipsformer	Puste inn de løse gips pulver og hudirritasjon av pulveren	1	A	A	A	A	A1	Bruk vernebrille og støvmaske.
5	Slip Casting	Puste inn de løse gips pulver og hudirritasjon av pulveren	1	A	A	A	A	A1	Bruk alltid vernebriller og en støvmaske.
#REF!	#REF!	Hvis man bruker varmelamper eller varmeovn, det er risiko for forbrenning	4	B	A	A	A	B4	Hvis man bruker varmelamper/varmeovn til å tørke mølde/deler, vil lampene være varme, så må man være forsiktig når man rundt varmelamper/varmeovn.

Appendix B: Popular science article

How does viscosity affect the stability of silicon carbide slip?

Written by Rongling Yuan.

Introduction of silicon carbide

Silicon carbide ceramics have high mechanical strength, high temperature resistance, wear resistance, corrosion resistance, small thermal expansion coefficient, high thermal conductivity, etc., and are often used in the manufacture of combustion chambers, high temperature exhaust devices, temperature resistant patches, aircraft engine components, chemical reaction vessel tubes



Figure 1: Silicon carbide [1]

and other mechanical components under severe conditions. Silicon carbide is a widely used advanced engineering material and the products have a broad market and application areas to be developed in the fields of energy, metallurgy, machinery, building materials and chemicals.

Silicon carbide ceramics are becoming more and more extensive. For satisfying the needs of industrial production, slip casting is used to produce silicon carbide products required by the market. This casting technique requires the preparation of silicon carbide slip with high solids content with low viscosity to achieve good slip stability [1].

Slip casting process

Casting Methods is more practical. It has many advantages such as simple equipment, uniform distribution of green body, fewer defects and more suitable to produce complex parts. Slip casting as shown in Figure 2 is the common method used to shape silicon carbide ceramic products. The suitable amount additives (such as wetting agents, dispersing agent or deflocculant) to dissolve the raw silicon carbide powder in the solution with a stable condition and prepared a slip. The slip is poured into the plaster mold which has the character of absorbing moisture from slip. After the water is absorbed by plaster mold, a uniform slip layer with a certain thickness is formed. During the dehydration and draining process without heat, a green body with a certain hardness is formed at this time and drained the slip out. The green body goes through the partial drying and separated from plaster mold. The heat is applied after the green body is shaped and goes through a sintering process. To improve the silicon carbide slip with good stability, it is necessary to have research on the viscosity of silicon carbide slip [2].



Figure 2: Slip casting process [2].

Viscosity research on silicon carbide slip

To research the viscosity of silicon carbide ceramic slip, the properties of silicon carbide slip need to know. Rheological properties changing the flow and deformation behavior of slip, dispersion mechanism of dispersant preventing particles approaching, and the effects of the slip properties on the green body which adjust the pH value in the slip can adjust the electric charge on the particles surface, so the electric charge increases, the electric double layer repulsion increases, and a stable slip state is achieved. Basic theory of controlling stabilized ceramic slip is obtained. Then, some experiments must be done to analyze the factors which affect the viscosity of silicon carbide slip, such as amount of dispersant and solid content.

The four different ratios of solid content, water and dispersant were prepared and measured with spindle nr. 63 of Brookfield viscometer.

Slip A contains 80 wt% solid content with 0.03 g AMP-dispersant, slip B contains 80 wt% solid content with 0.12 g AMP-dispersant, slip C contains 87 wt% solid content with 0.03 g AMP-dispersant, and slip D contains 87 wt% solid content with 0.12 g AMP-dispersant. After viscosity measurement, the results as shown in Figure 3.

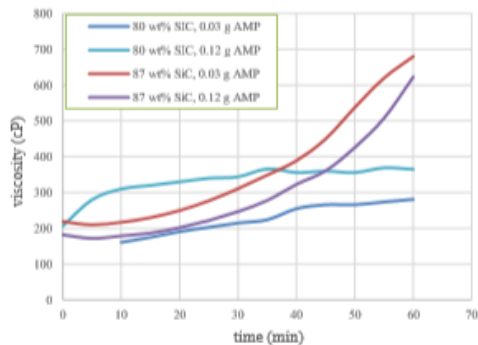


Figure 3: The viscosity as a function of time.

During the viscosity measurement, observed that the slip was precipitated over time. The longer the time, the more obvious the precipitation phenomenon, and the thicker the precipitation layer at the bottom of the slip. Slip A and B measurements were first completed, slip C and D were measured one week after. Observed that the

slip A and B were precipitated, after the hard hand stirring, the original slip state was restored. But the sediment layer of slip C and D, were solidified into a hard block with ca. 1 cm thick at the bottom of the bottle. The precipitation phenomenon in the slip could lead to the upper layer of slip got thinner over time, and if the depth of spindle also in the upper layer, so a decreased viscosity as a result. Similarly, the bottom layer of the slip became more and more viscous by particle settling, and the spindle depth is in the slip's lower layer, which also caused the viscosity increased or over the test range. The Figure 3 shows that the stability of the slip A and B has further improved with the 80 wt% solid content, there is not much change in the viscosity value over the entire range of dispersant from 0.03-0.12 g because of the flocculated layer formed during the time. the slip A and B with same 80 wt% solid content, the stability of the slip with 0.03 g of dispersant was the best. But in the same 87wt% solid content, slip C and D, with 0.12g of dispersant had the best stability.

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