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REVIEW ARTICLE

A Review of Viscosity Measurement Techniques for Semi-Solid Metal Fluids in Thixoforming Processes

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ABSTRACT - This paper critically reviews two viscometry techniques: capillary viscometers and parallel plate compression (PPC) viscometers, which are used in the viscosity measurement of semi-solid metal (SSM) fluids for thixo routes. Capillary and PPC viscometers have emerged as valuable viscometers in the study of thixo routes of SSM, with insights into their unique flow behavior being explored. Thixoprocessing of SSM is particularly suitable for specific alloys, especially those with specific solidification properties such as eutectics. It is a method of thixoforming that uses a material's non-dendritic microstructure. The thixo method's viscosity measurement is challenging, requiring sample preparation, sophisticated equipment, precise temperature, and pressure control. Previous studies have lacked sufficient information on measurement methods, viscometers, and limitations to measuring viscosity for the thixo route of SSM. Hence, this study thoroughly examines the principles, advantages, limitations, and application areas of viscometry methods. Several factors influencing viscosity measurements, such as temperature, shear rate, and sample preparation, are discussed in detail. A comparative analysis is conducted to evaluate the performance and accuracy of capillary viscometers and parallel plate compression viscometers. This review will provide a comprehensive understanding of SSM's viscometry techniques and assist researchers and practitioners in selecting the most appropriate method for their specific applications in semi-solid metal processing and characterization.

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Semi-solid metals

1. INTRODUCTION

Semi-solid metal (SSM) processing is a technology that has a wide potential for producing alloys within the solidification range as an alternative to the traditional die-casting process [1]. Numerous studies have demonstrated that SSM processing can lead to improved mechanical properties, reduced porosity, and enhanced formability compared to conventional solid or liquid metal processing methods [2], [3], [4]. Furthermore, this technology has found applications in a wide range of industries, from automotive and aerospace to electronics and medical devices, driving ongoing investigations into the optimization of SSM processing techniques and their integration into industrial manufacturing practices [5].

The flow behavior of SSM is a crucial aspect of determining and optimizing their processing. The Rheo route and the Thixo route are two advanced processing methods used in SSM casting, a specialized technique within the field of metal casting [6], [7]. The Rheo Route method involves carefully controlling the temperature and stirring rate of a molten metal or alloy to induce a semi-solid state with a globular or spheroidal microstructure. In the Rheo Route, the material is typically in a semi-solid state with a higher fraction of liquid compared to the Thixo route. This results in a lower viscosity for materials in the Rheo route, making them more flowable and suitable for processes like die-casting and injection molding. The semi-solid slurry in the Rheo Route is often likened to a thick, viscous paste [8]. The thixo route method focuses on creating semi-solid feedstock materials by manipulating the solid fraction and morphology of metal or alloy slurries through a combination of cooling and mechanical processing. In the Thixo route, the material is semi-solid, with a higher fraction of solid particles suspended in the liquid matrix. This higher solid fraction gives Thixo materials a higher viscosity compared to Rheo materials. This increased viscosity is beneficial for processes where better mold filling and maintaining the shape of intricate parts are crucial. The thixo route has been found to help improve mechanical properties over the rheo route [9],[10].

The viscosity of SSM is vital for achieving high-quality and cost-effective manufacturing processes [11]. Rheo route viscosity is a general concept related to the viscosity of materials in rheological studies, while thixo route viscosity refers specifically to the initial viscosity of thixotropic materials. Better viscometers are needed to describe and analyze in detail how materials flow and deform under different conditions [12]. Among the different viscometer methods, capillary viscometry and parallel plate compression (PPC) viscometry have emerged as prominent tools for investigating the rheological properties of thixo route SSM [13]. A capillary viscometer involves the measurement of the flow of SSM

through a capillary tube, while a PPC viscometer employs the compression of SSM between two parallel plates. These techniques offer valuable insights into the viscosity, shear thinning behavior, and flow mechanisms of SSM [14]. However, a critical evaluation and comparative analysis of these viscometer methods for semi-solid metals are necessary to understand their strengths, limitations, and suitability for different applications.

Various literatures provide details on viscosity and viscometry methods. However, the research gap lies in the absence of a comprehensive and critical analysis that compares capillary viscometer and PPC viscometer techniques in terms of their applicability, accuracy, and reliability for the viscosity characterization of semi-solid metals [15], [16]. Furthermore, a thorough comparative review is needed to identify their relative advantages and limitations, their sensitivity to various factors affecting viscosity measurements, and their suitability for different types of SSM applications. By addressing this research gap, we can gain valuable insights into the strengths and weaknesses of capillary viscometry and PPC viscometry techniques when applied to SSM. This knowledge will help researchers, engineers, and material scientists make informed decisions regarding the selection of the most appropriate viscometry method for their specific SSM research or industrial needs. Additionally, a critical review of the existing literature can highlight any knowledge gaps and provide recommendations for future research directions in SSM viscometry, leading to advancements in process optimization, material design, and industrial applications of semi-solid metals.

This paper aims to provide a comprehensive and critical review of SSM capillary viscometers and PPC viscometers for the thixo routes. It will discuss the underlying principles, measurement techniques, advantages, and limitations of each method. Furthermore, the article will delve into the factors influencing viscosity measurements, including temperature, shear rate, and sample preparation, as well as how they impact the accuracy and reliability of the viscometry results. For SSM, two commonly used viscometry techniques are capillary viscometry and parallel plate compression viscometry. Although these methods are widely used, a critical review comparing the strengths, limitations, and suitability of capillary viscometry and PPC viscometry will address this gap in the existing literature.

2. RHEOLOGICAL BEHAVIOR OF SSM

SSM is a class of materials that exhibit a unique rheological behavior characterized by a mixture of solid and liquid phases. Unlike conventional solid metals, SSM possesses a thixotropic or shear thinning behavior, allowing them to flow under applied stress while maintaining a stable microstructure [17], [18]. Semi-solid metal applications continue to grow as researchers and industries investigate their unique properties and develop new processing methods. With their versatility, enhanced mechanical properties, and lightweight design, semi-solid metals offer innovative solutions in diverse industries [19]. Characterization of SSM involves assessing various properties and microstructural features to understand and optimize the material's behavior and performance. Characterization of semi-solid metals combines various analytical techniques to evaluate microstructural features, chemical properties, thermal behavior, mechanical and electrical properties, dimensional stability, and chemical composition [20]. These characteristics enable a comprehensive understanding of SSM and help improve its implementation and application-specific requirements.

The rheological behavior of SSM is important for processing and forming applications. Rheological characterization involves measuring flow behavior, viscosity, shear stress, and shear rate dependencies [21]. The rheological behavior of semi-solid metals (SSM) refers to their flow characteristics under applied stress or shear conditions. Unlike conventional solid metals, SSM exhibits a unique thixotropic behavior, wherein their viscosity decreases with increasing shear rate. This behavior is caused by the presence of a non-equilibrium microstructure consisting of solid particles suspended in a partially molten metal matrix [22]. Various factors, including temperature, solid fraction, particle size distribution, alloy composition, and processing history, influence the rheological behavior of SSM. Understanding and characterizing the rheological properties of SSM is crucial for optimizing processing conditions and achieving desired material properties.

2.1 Flow Behavior

SSM exhibits shear thinning or pseudoplastic behavior, where the apparent viscosity decreases as the shear rate increases. This means that SSM flows more easily under higher shear rates, facilitating their processing and shaping. The flow behavior can be quantified using rheological tests, such as shear rate sweeps or flow curves, which measure the relationship between shear stress and shear rate. Several factors have been identified that influence SSM's flow behavior [23]. Temperature plays a significant role in affecting solidification kinetics and the viscosity-temperature relationship of the material. The solid fraction and particle size distribution also have an impact on SSM's flow properties, with higher solid fractions leading to increased viscosity and particle interactions influencing flow behavior [24]. Moreover, the alloy composition, including the types and concentrations of alloying elements, influences the flow characteristics of SSM. The review also highlights the influence of processing techniques and history on the flow behavior of SSM. Various processing methods, such as thixoforging, rheocasting, and semi-solid metal injection molding, have been investigated for their impact on the flow properties of SSM [6]. Additionally, the heating and cooling rates during processing have been found to affect SSM's microstructure and flow behavior.

2.2 Viscosity

The viscosity of SSM is a key rheological property that determines their flow resistance [25]. At low shear rates, SSM exhibits high viscosities, indicating their resistance to flow. Several factors, including temperature, pressure, solid fraction, alloy composition, and shear rate, influence the viscosity of SSM. Temperature has a significant impact on SSM

viscosity [26]. As temperature increases, viscosity typically decreases due to the reduction in the solid fraction and the increased mobility of the liquid phase. In SSM, the temperature dependence of viscosity can follow an Arrhenius relationship, where the viscosity decreases exponentially with temperature. The solid fraction, which represents the volume fraction of solid particles in the SSM matrix, affects its viscosity. Higher solid fractions generally result in higher viscosities, indicating greater resistance to flow. The arrangement and distribution of solid particles influence the viscosity behavior, as particle interactions can increase the effective viscosity [27]. The alloy composition, including the type and concentration of alloying elements, can have a significant impact on SSM viscosity. Alloying elements may influence the solidification process, the morphology of solid particles, and the liquid phase characteristics, affecting the overall viscosity behavior. The viscosity of SSM's thixotropic behavior with time and shear rate is significant, and its schematic diagram is presented in Figure 1. Different alloy compositions can exhibit distinct viscosity profiles, offering opportunities for tailoring material flow properties. As the shear rate increases, the viscosity decreases significantly due to the breakdown of the particle network and the realignment of solid particles in the liquid matrix [28].

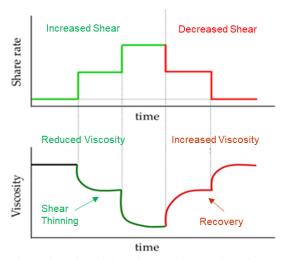


Figure 1. Schematic diagram of the viscosity of the thixotropic behavior of SSM with time and shear rate [29]

Figure 1 illustrates the viscosity of SSM's thixotropic behavior with time and shear rate. Shear thinning behavior is a characteristic feature of SSM, wherein the apparent viscosity decreases with increasing shear rate. This behavior is attributed to the disruption of the solid particle network and the shear-induced orientation and alignment of solid particles. Shear thinning facilitates better flowability and mold filling during processing, enabling complex shapes to be formed with reduced pressure requirements [30]. SSM may exhibit yield stress, which represents the minimum stress required to initiate flow. Below the yield stress, the material behaves as a solid, resisting deformation. However, once the yield stress is surpassed, the material transitions into a flow state. Factors such as solid fraction, particle size, and interparticle interactions influence the yield stress. SSM are often strain rate sensitive, meaning their flow behavior depends on the rate of deformation. Higher strain rates result in reduced viscosity and improved flowability [31]. It is important to consider this strain rate sensitivity when optimizing processes and determining the appropriate processing conditions for forming SSM. Understanding the rheological behavior of SSM is essential for designing and optimizing processing techniques such as thixo forging, rheo casting, or semi-solid metal injection molding. By controlling the rheological properties of SSM, manufacturers can achieve improved material flow, reduced defects, and enhanced mechanical properties in the final components [32].

3. VISCOMETERS

Viscometers are instruments that measure fluid viscosity. They play a crucial role in various industries and research fields where viscosity determination is essential for process control, quality assurance, and material characterization [33]. Several types of viscometers are available, each employing different principles and techniques for viscosity measurement. Viscometers can be classified based on various factors, including the principle of operation, the nature of the fluid being tested, and the specific application requirements. There are some common classifications of viscometers [13].

3.1 Principle of Operation

Viscometers are instruments used to measure the viscosity of fluids, and they operate based on various principles. They are divided into six types based on the principle of operation.

Rotational Viscometers: These viscometers measure viscosity by rotating a spindle or rotor in the fluid and measuring the torque required to overcome the resistance to rotation. According to M. Modigell's studies, rotational viscometers consisting of concentrically arranged cylinders are most commonly used for studying semi-solid metals. In a Couette-type viscometer, the cup rotates, and the bob is stationary, whereas in a Searle viscometer, the bob rotates, and the cup is stationary. During the test, the specimen is cut in the space between them. The shear stress in the wall is related to the torque [34]. The cone and plate viscometer excels at high temperatures in the cyclic measurement technique for estimating

the viscosity of liquid metals and alloys [35]. Vane-shaped viscometers evaluate the low-strain modulus and steady-state flow curves of structured fluids and have many advantages, such as simplicity, ease of cleaning, and, above all, the elimination of extreme wall-slip effects [36]. Figure 2. shows a schematic diagram of rotational viscometers, as well as their types.

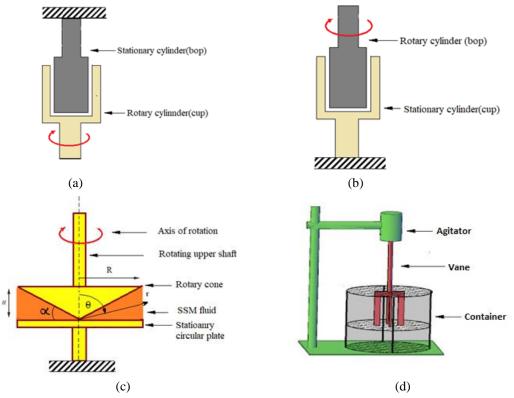


Figure 2. Schematic diagram of rotational viscometers: (a) Couette type viscometer [37], (b) Searle type viscometer [37], (c) Cone and plate viscometer [38], and (d) Vane type viscometer [39]

Capillary Viscometers: A. Grupi's studies show that capillary viscometers, commonly utilized in laboratories, measure fluid viscosity by timing the flow of a liquid through a narrow tube under gravity or applied pressure, adhering to Poiseuille's law. Instruments such as the Ostwald, Ubbelohde, and Cannon-Fenske viscometers are key types, each designed to enhance accuracy for specific applications. They are widely valued for their simplicity, precision, and cost-effectiveness. Despite being primarily suited for low- to moderate-viscosity Newtonian fluids, capillary viscometers remain indispensable due to their straightforward operation and reliable measurements. J. Cheng. et al. explained that capillary viscometers measure viscosity by measuring the flow rate of a fluid through a capillary tube under a specific pressure or gravity [40]. A schematic diagram of a capillary viscometer for high-fraction solid fluids is presented in Figure 3.

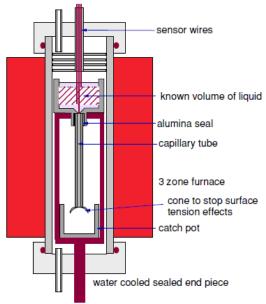


Figure 3. Schematic diagram of capillary viscometer for high fraction solid fluids [41]

Vibrational Viscometers: Vibrational viscometers measure fluid viscosity by detecting the damping effect on a vibrating sensor, such as a rod or tuning fork, immersed in the fluid. The damping, which is influenced by the fluid's viscosity, alters the sensor's vibration frequency or amplitude. These viscometers are highly valued for their ability to provide continuous, real-time measurements, making them ideal for process control in industrial settings. Vibrational viscometers measure viscosity by determining the fluid's damping effect on the vibration of a probe or resonating element [42].

Falling Ball Viscometers: Falling ball viscometers, also known as falling sphere viscometers, measure the viscosity of a fluid by analyzing the motion of a sphere as it falls through the fluid under the influence of gravity. The primary principle behind this type of viscometer is Stokes' law, which relates the terminal velocity of a sphere moving through a viscous medium to the viscosity of that medium. Falling ball viscometers are valuable tools for measuring fluid viscosity, offering a balance of simplicity, accuracy, and cost-effectiveness. Their use across various industries highlights their importance in ensuring product quality and consistency. Despite some limitations, such as sensitivity to temperature and challenges with opaque fluids, falling ball viscometers remain a trusted method for viscosity measurement in many applications. Falling ball viscometers determine viscosity by measuring the time taken for a ball to fall through a fluid-filled tube [43]. A schematic diagram of the falling ball viscometer is presented in Figure 4.

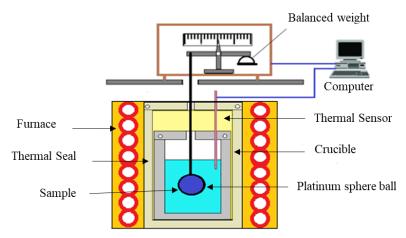


Figure 4. Schematic diagram of falling ball viscometer [13]

Oscillatory Viscometers: Oscillatory viscometers, also known as oscillatory rheometers, measure the viscosity of fluids by analyzing the damping effect on an oscillating sensor immersed in the fluid [44]. The sensor, often a disc or a cylinder, is set into oscillatory motion, and the fluid's viscosity is determined by the resistance it offers to this motion. The degree of damping, which affects the amplitude and frequency of the oscillations, is directly related to the fluid's viscosity. Oscillatory viscometers measure the response of a sample to oscillatory shear deformations and provide information on complex rheological properties, such as viscoelasticity [45]. A schematic diagram of the oscillatory plate viscometer is presented in Figure 5.

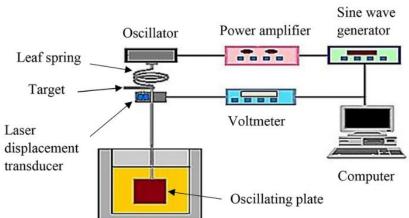


Figure 5. Schematic diagram of oscillatory plate viscometer [13]

• **Compression viscometers:** A compression viscometer measures viscosity by measuring the deformation or shrinkage that results from applying a controlled force or pressure to a sample [46].

Nature of the Fluid: Based on the nature of the fluid being measured, viscometers can be classified into Newtonian viscometers and non-Newtonian viscometers.

• **Newtonian viscometers:** These viscometers are suitable for measuring the viscosity of fluids that exhibit constant viscosity regardless of the shear rate, such as water or some oils [47].

• **Non-Newtonian viscometers:** Non-Newtonian viscometers are designed to measure the viscosity of fluids that exhibit shear rate-dependent viscosity, such as polymer solutions, suspensions, and emulsions [48].

Application-Specific Classification: Viscometers can be classified based on their suitability for specific applications or industries due to variations in viscosity range, sample volume, and environmental conditions.

- **Process viscometers:** These viscometers are designed for real-time or online viscosity monitoring in industrial processes, providing continuous measurements to ensure process control and quality assurance.
- Laboratory viscometers: Laboratory viscometers are more versatile and precise instruments used for detailed viscosity characterization, often in research and development settings [49].
- **Portable viscometers:** Portable viscometers are hand-held or compact devices suitable for on-site or field measurements, offering convenience and ease of use.

Measured Parameters: Viscometers can be classified based on the specific parameters they measure.

- Absolute viscometers: Absolute viscometers directly measure the viscosity of the fluid.
- **Relative viscometers:** Relative viscometers compare a fluid's viscosity to a reference fluid, providing a relative measure of viscosity without absolute values.

Sensor Technology: Based on the sensor technology used to measure viscosity and related properties, viscometers are classified as,

Mechanical Viscometers: Mechanical viscometers are instruments used to measure the viscosity of fluids. Viscosity is a property that describes a fluid's resistance to flow. Mechanical viscometers rely on physical mechanisms and principles to determine the viscosity of a fluid sample. Mechanical viscometers rely on mechanical means, such as rotational or oscillatory motion, to measure viscosity.

Electronic Viscometers: Electronic viscometers are sophisticated instruments used to measure the viscosity of fluids using electronic and digital technologies. Unlike traditional mechanical viscometers, which rely on physical principles and manual observation, electronic viscometers use sensors, digital displays, and automated systems to provide precise and repeatable measurements. Electronic viscometers use electronic sensors, transducers, or resonators to measure viscosity based on changes in electrical or mechanical properties [42].

It's important to note that these classifications are not mutually exclusive, and a viscometer may belong to multiple categories depending on its design and capabilities. The classification of viscometers based on various parameters, including their principle of operation, nature of the fluid, measured parameters, and sensor technology, reveals a diverse array of instruments tailored to specific applications. This diverse classification underscores the critical role viscometers play in understanding and optimizing fluid behavior, ensuring product quality, and advancing research and development across numerous fields. Capillary and PPC viscometers are important tools for measuring the viscosity of SSM due to their ability to handle non-Newtonian behavior, their suitability for non-homogeneous materials, and their standardized measurement methods. These viscometers provide valuable information about SSM's flow properties and rheological behavior, enabling better understanding and control of these materials in various industries.

4. CAPILLARY VISCOMETERS

Professor Morris Cohen, an American physicist and metallurgist, is credited with inventing the capillary tube viscometer for measuring the viscosity of semi-solid metals. Cohen developed the capillary tube method, also known as the "Cohen method," as a means to measure the viscosity of molten metals and alloys in the 1940s [50]. The capillary tube viscometer works by measuring the flow rate of a semi-solid metal through a capillary tube under the influence of gravity or applied pressure. The material's viscosity can be determined by examining the relationship between flow rate and applied force [14]. This method is particularly useful for high-temperature materials like molten metals, where traditional viscometers are not suitable [51].

Capillary viscometers are widely used for viscosity measurements and rely on the flow of a fluid through a capillary tube under specific pressure or gravitational forces [52]. According to D. K. Hilton's studies, capillary viscometers are based on the principle of Poiseuille's law, which states that the flow rate of a Newtonian fluid through a capillary tube is directly proportional to the pressure drop and inversely proportional to the viscosity and length of the capillary [53]. Furthermore, it operates on the laminar flow principle, which is smooth and parallel to the capillary walls. Viscosity is determined by measuring the flow rate under a specific pressure or gravitational force, which is related to the fluid's resistance due to its viscosity [15]. Figure 6 shows a schematic diagram of a capillary viscometer that measures viscosity in both the high liquid state and the high solid state.

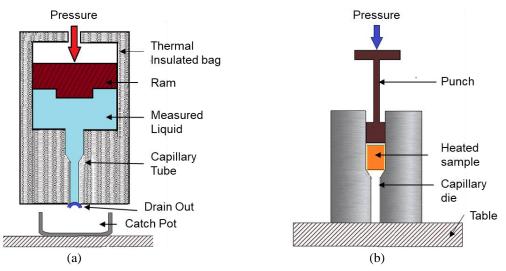


Figure 6. Schematic diagram of capillary viscometer for the (a) high liquid phase, and (b) high solid phase [13]

A. Gruby et al. used a fully automated calibration capillary viscometer to calculate the concentration and shear dependence of macromolecular solutions' viscosity. Capillary viscometers involve the following steps: A measured volume of the fluid sample is introduced into the capillary tube. The capillary tube is placed vertically or horizontally, and gravity or controlled pressure is applied to initiate flow [54]. The time taken for the fluid to flow through a certain capillary length is measured. The flow time is used to calculate the fluid's viscosity based on the capillary's dimensions and the known pressure or gravitational force. According to the Hagen-Poiseuille equation (1), the time taken for the fluid to flow through a certain capillary length is measured. The flow time is used to calculate the fluid's viscosity based on the capillary's dimensions and the known pressure or gravitational force. According to Hagen-Poiseuille's equation (1) [55].

$$\mu = \frac{\Delta P \pi R^4}{8LQ} \tag{1}$$

where is the viscosity, P is the pressure drop along the length of the capillary, R is the radius of the tube, L is the length of the capillary, and Q is the flow rate.

Capillary viscometers offer a simple method for viscosity measurements. They can be used for a wide range of fluids, including Newtonian and non-Newtonian fluids. Capillary viscometers provide measurements at different shear rates, allowing for the evaluation of shear rate dependence on viscosity [56]. Capillary viscometers may not be suitable for highly viscous or non-Newtonian fluids that exhibit significant flow resistance or complex flow behavior [55]. Accuracy can be affected by uncertainties in capillary dimensions, temperature, and pressure measurements. Cleaning and preparing the capillary tubes can be time-consuming and require careful attention to avoid contamination or blockage [57]. Capillary viscometer measurements should focus on temperature, shear rate, and sample preparation. Viscosity is highly temperature-dependent, and capillary viscometers should be operated at controlled temperatures to ensure accurate measurements. Temperature variations can have a significant impact on the viscosity, and corrections or calibration factors may be required [58]. Capillary viscometers typically operate at low shear rates. It is important to consider that viscosity may vary with shear rate, especially for non-Newtonian fluids. Understanding the shear rate conditions and ensuring they are appropriate for the specific fluid being tested is crucial [48]. The condition of the fluid sample, such as cleanliness and homogeneity, can influence viscosity measurements. Any impurities or contaminants present in the sample may alter the viscosity, and careful sample preparation is essential for accurate results [59]. Capillary viscometers offer a reliable method for viscosity measurements, particularly for Newtonian fluids. Understanding the principles, advantages, and limitations of capillary viscometers is important for selecting the appropriate instrument and interpreting the results accurately. Considering factors like temperature, shear rate, and sample preparation is crucial for obtaining reliable viscosity measurements using capillary viscometry [60].

Efforts have been made to establish calibration standards and procedures to ensure consistent and accurate viscosity measurements. Calibration involves determining the relationship between flow rate and viscosity using reference materials with known viscosities [49]. These standards help to reduce measurement errors and increase the viscometer's reliability. Maintaining precise temperature control is crucial when measuring the viscosity of semi-solid metals [61]. Temperature variations can have a significant impact on the flow properties of the material as well as the viscosity measurement [62]. Advanced capillary tube viscometers require temperature control systems to ensure that the sample remains at a constant temperature during testing [63]. Ensuring a uniform temperature distribution and avoiding temperature gradients within the sample is crucial to obtaining accurate viscosity values. The viscosity of semi-solid metals often exhibits a strong dependency on shear rate or applied stress [64]. The capillary tube viscometer typically operates at specific shear rates, but expanding the results to other shear rates may reveal uncertainty. Heat loss during the measurement process can affect the viscosity values, necessitating efficient insulation and temperature control

mechanisms [65], [66]. The capillary tube viscometer is primarily designed to measure the viscosity of semi-solid metals and may not be suitable for all types of materials. Different alloys or non-metallic substances may require alternative viscometer designs or techniques [67]. The advantages and disadvantages of the capillary viscometer are provided in Figure 7.

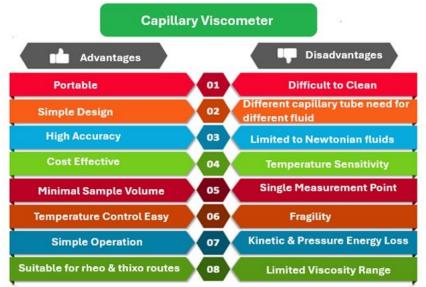


Figure 7. Advantages and disadvantages of capillary viscometer

The capillary tube viscometer's advantages and disadvantages are categorized based on the challenges reported by most researchers [68], [69], [70]. Understand the flow behavior of semi-solid metals, as well as progress in viscometer design and calibrated instruments. It helps to improve the accuracy and applicability of the capillary tube viscometer for measuring SSM viscosity.

5. PARALLEL PLATE COMPRESSION VISCOMETER

Parallel Plate Compression (PPC) viscometers, also known as plate-plate viscometers or squeeze flow viscometers, are commonly used instruments for measuring the viscosity of fluids [71]. It operates on the principle of applying controlled pressure to a sample confined between two parallel plates and measuring the fluid's resulting deformation or flow behavior [16], [72]. P. O. Charreyron et al. studies have relied on the principles of viscous flow and deformation under stress while investigating the rheological properties of semi-solid dendritic Sn-Pb alloys at low strain rates. Furthermore, the fluid's viscosity determines the rate of flow or deformation, and the viscosity can be calculated based on the observed response by applying a known pressure [73]. Figure 8 presents a diagram of a parallel plate compression viscometer for measuring the viscosity of semi-solid metals.

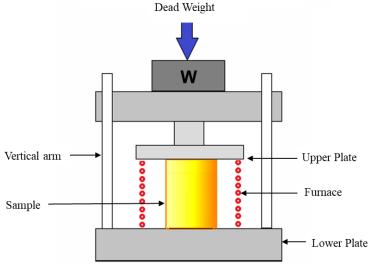


Figure 8. Schematic diagram of parallel plate compression viscometer [74]

In Y. Fukui's evaluation study of the deformation behavior of semi-solid hypereutectic Al-Si alloy, a small volume of liquid sample is placed between two parallel plates. The plates are brought together to establish a defined gap or applied pressure. The fluid's deformation, or flow, is measured under applied pressure [75]. The flow characteristics, or deformation data, are used to determine the fluid's viscosity. Equations (2) to (4) are provided to measure the viscosity of

semi-solid metals using a parallel plate compression viscometer [46]. In Lashkari's study, the viscosity of the Al-Si alloy in the semi-solid condition was determined using a plate compression viscometer.

$$Initial Pressure(P_0) = \frac{Fh_0}{V}$$
 (2)

Compression Rate (CR) =
$$\frac{\Delta_h}{\Delta_t}$$
 (3)

$$\frac{3Vh_0}{8\pi P_0} \left(\frac{1}{h^4} - \frac{1}{h_0^4} \right) = \frac{t}{\mu} \tag{4}$$

where μ is calculated viscosity, the applied force on the semi-solid billet is F, the initial pressure is P₀, the volume of the semi-solid slurry is V, the instantaneous height of the billet is h, and time is t, the change in height (Δ h), and the change in time (Δ t) [76].

PPC viscometers allow for measurements at various shear rates by adjusting the applied pressure or the plate gap. It is suitable for both Newtonian and non-Newtonian fluids, as the measurement can be tailored to the sample's specific flow behavior. In A. Blouri's study of the microstructure and rheological properties of semi-solid 7075 emulsions, PPC viscometers provide good accuracy and repeatability, making them reliable instruments for viscosity measurements [77]. Factors such as uncertainties in pressure measurements, temperature variations, and the sample's non-uniformity between the plates influenced the accuracy of measurements. PPC viscometers accurately capture the flow behavior of highly viscous fluids, as well as those with complex rheological properties. Sample preparation and handling required attention to ensure proper filling of the gap between the plates and avoid air entrapment or other artifacts that could affect the measurement [78].

The PPC viscometer plays an important role in the optimal methods for measuring SSM low viscosity [79]. Several researchers have successfully studied semi-solid metals using a parallel-plate compression viscometer at specific solid fractions and low shear rates [73], [80]. It can detect initial pressure, and shear rate in the early stages of rapid compression. In 2002, Yurko and Flemings at MIT introduced a drop-forge viscometer with parallel plates to determine viscosity by compressing a sample at speeds less than 10 s and calculating shear rate and stress [81]. In this test, the sample was compressed using a dead weight. In 2006, Lashkari and Gomashchi developed a new PPC viscometer that measures viscosity by compressing a sample with dead weight and pneumatic force [74]. It calculated the viscosity as a function of the deformation versus the time the sample was compressed during the compression test. The advantages and disadvantages of PPC viscometers are provided in Figure 9.

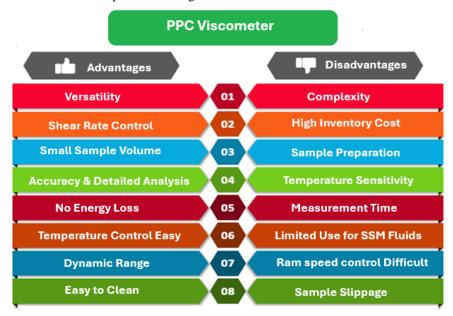


Figure 9. Advantages and disadvantages of parallel plate compression (PPC) viscometer

The viscosity of most fluids is highly temperature-dependent. Accurate temperature control is required during viscosity measurements to account for the sample's temperature sensitivity. To account for temperature variations, corrections or calibration factors may be required [13]. In parallel plate compression viscometry, shear rates can be adjusted by altering the applied pressure or the plate gap. Understanding the shear rate conditions and their impact on the viscosity of the specific fluid being tested is important for proper interpretation of the results. The sample should be homogeneous and free from air bubbles or other contaminants that may affect the flow behavior and introduce errors in the viscosity measurement [82]. For reliable results, it is critical to ensure proper sample preparation, fill the gap between

the plates, and minimize any sample handling disturbances. PPC viscometers offer a versatile method for measuring fluids' viscosity, suitable for Newtonian and non-Newtonian samples. Understanding parallel plate viscometry's measurement principles, advantages, and limitations is crucial for selecting the appropriate instrument and ensuring accurate viscosity measurements. R. Meshkabadi's studies suggest that factors such as temperature, shear rate, and sample preparation are essential to obtaining reliable viscosity data using PBC viscometry [83].

6. CRITICAL ANALYSIS

Capillary viscometers and PPC viscometers are two commonly used techniques for viscosity measurements in various fluids, including semi-solid metals (SSM). While both methods aim to provide insights into the flow behavior and rheological properties of materials, they differ in their measurement principles, experimental setups, and applicability [67]–[70]. Table 1 presents the similarities and differences between capillary viscometers and PPC viscometers.

Table 1. Presents the comparison between the Capillary viscometer and PPC viscometer

| Capillary Viscometer | PPC Viscometer | Cite from |
|---|--|--------------|
| Viscosity is measured by observing the flow of a liquid through a capillary tube under a certain pressure or gravity. | Viscosity is measured by measuring the stress exerted on a sample compressed between two parallel plates. | [64] |
| Determined based on the flow rate and the dimensions of the capillary tube. | Determined based on the compression rate and the shear rate. | [49] |
| Measured the Newtonian and non-Newtonian fluids., but mostly suitable for Newtonian fluids. | Measured the Newtonian and non-Newtonian fluids., but mostly suitable for non-Newtonian fluids. | [53] |
| Suitable for low to medium viscosity fluids | Suitable for studying the rheological behavior and shear-thinning properties of materials including semisolid metals. | [57] |
| Accuracy is poor when measuring non-Newtonian fluids with complex flow behavior | Good accuracy and sensitivity for non-Newtonian fluids with shear rate-dependent viscosity. | [88] |
| The presence of bubbles or impurities in the sample will affect flow behavior and measurement accuracy | The presence of impurities does not affect flow behavior and measurement accuracy | [89] |
| The size or shape of the sample should be measured before measurement | Does not required | [90] |
| Suitable for a wide range of fluids and allows for measurements at different shear rates. | The rheological behavior of a high solid-state material at different shear rates can be accurately measured in rapid measurements. | [68] |
| Maximum temperature range of about 1200 °C | A temperature range above 1200 °C can also be measured | [91] |
| Errors are caused by wall effects and frictional losses | Errors are caused by ram speed, billet slip, and calibration problems | [92] |

Viscosity is measured based on the time it takes for a liquid to flow through a capillary tube. The radius and length of the capillary are responsible for the kinetic energy effect of SSM. Different viscometers are required depending on fluidity, temperature, and pressure effects [40], [92], [93]. Several problems exist when measuring fluid volume, pressure, and temperature before entering highly viscous SSM fluids into the capillary. It can be used both as a single-point viscometer and a multi-point viscometer [34], [94]. Capillary radius and length, shear stress, shear rate, flow rate, pressure drop, and frictional loss have significant effects on the measurement. A stable, uniform laminar flow can be obtained by using a long capillary tube. Preferably, the ratio between the inner diameter and length of the capillary tube is greater than 1:65 [65], [91]. Increasing the applied pressure will produce a specific effect, but it is subject to limitations. This can increase turbulent flow or wall effects [95]. A critical analysis of capillary viscometers and PPC viscometers compiled from various literatures is presented in Table 2.

The PPC viscometer provides a controlled and well-defined shear flow between the plates, allowing for accurate viscosity measurements. This viscometer is suitable for measuring the viscosity of both low-viscosity liquids and highly viscous materials, making it versatile for a wide range of applications. The PPC viscometer can handle both Newtonian fluids, which exhibit linear viscosity behavior, and non-Newtonian fluids, which have complex viscosity behaviors. It is capable of measuring both steady-state and dynamic viscosity, providing insights into the flow behavior of fluids under different conditions. The viscometer allows for the measurement of various sample sizes, ranging from small quantities to larger volumes, depending on the design and specifications of the instrument. With proper calibration and setup, the parallel plate compression viscometer can provide accurate and precise viscosity measurements, enabling reliable data analysis and quality control.

The PPC viscometer is sensitive to temperature changes, which can affect the viscosity measurements. Temperature control and correction methods are often necessary to ensure accurate results. Compared to other viscometer types, such as capillary viscometers, the PPC can be more complex in terms of design and operation. This complexity may require a higher initial investment and expertise in handling the instrument. Achieving ideal conditions, such as fully developed flow profiles and negligible edge effects, can be challenging in practice. Corrections and modifications may be necessary to account for these deviations, especially when dealing with non-Newtonian fluids. The shear rates achievable in a PPC viscometer may be limited by the instrument's dimensions and setup. For certain fluids with extreme shear rate requirements, alternative viscometer designs or methods may be more suitable. Problems with ram speed and sample slip should be resolved.

Table 2. Critical analysis of capillary viscometer and PPC viscometer

| Aspect | Capillary Viscometer | PPC viscometer | Cite from |
|-------------------------------------|---|--|---------------|
| Principle | Flow rate through a narrow capillary tube | Resistance to flow between two parallel plates | [51] |
| Design | Simple and portable | Complicated and non-portable | [96] |
| Applicable Fluids | Newtonian and non-Newtonian | Newtonian and non-Newtonian | [74] |
| Preferable Method | Rheo and thixo routes | Thixo routes | [97] |
| Cost | Less | Reasonable | [68] |
| Losses | Kinetic energy, Pressure, Wall end effect | Surrounding Temperature | [48], [74] |
| Chemical reaction | Oxide inclusions | Nil | [14] |
| Premeasurement | Needed | No need | [91] |
| Cleaning | Difficult | Easy | [74] |
| Viscosity Index | 10^{-4} to 10^{-6} | $10^1 \text{ to } 10^9$ | [13] |
| Type | Single point & Multi point | Multi point | [13] |
| Suitable | More Liquid phase | More solid phase | [98] |
| Range of Viscosity | Wide range, suitable for low to high viscosities | Wide range, suitable for low to high viscosities | [99] |
| Measurement | Flow rate and pressure difference | Force required to maintain constant flow rate | [100] |
| Shear Rates | Dependent on capillary dimensions | Controlled and well-defined shear rates | [101] |
| Accuracy and Precision | Moderate to high, depending on setup and design | Moderate to high, depending on setup and design | [81] |
| Temperature Sensitivity | Sensitive to temperature variations | Sensitive to temperature variations | [57] |
| Rheological Behavior Measurement | Limited, primarily measures steady- state flow | Can measure steady-state and dynamic viscosity | [34] |
| Application Area | Chemical, pharmaceutical, and petroleum industries; research laboratories | Polymer processing, food and beverage production, cosmetics, research laboratories | [51] |

7. CONCLUSIONS

In conclusion, the PPC viscometer and the capillary viscometer are both helpful instruments for figuring out the viscosity of SSM fluids, although they have specific advantages and disadvantages of their own.

| Viscometer Type | Advantages | Limitations | Best Suited For |
|---------------------------------------|---|--|---|
| Capillary viscometer | Simple, cost-effective, wide viscosity range, versatile | Limited to steady-state flow, may need temperature control | • |
| Parallel plate compression viscometer | Controlled shear flow, measures steady-state and dynamic viscosity, broad capabilities | Sensitive to temperature, complex, costly | In-depth rheological studies for high-fraction solids |

- The capillary viscometer can handle a wide range of viscosities and is suitable for both Newtonian and non-Newtonian fluids. However, its measurements are primarily limited to steady-state flow and require temperature control for accurate results.
- The PPC viscometer can measure both steady-state and dynamic viscosities, providing insights into the flow behavior of fluids under different conditions. It is sensitive to temperature variations and is more complex and expensive compared to a capillary viscometer.
- Choosing between the two viscometers depends on the specific requirements of the application. The simplicity and versatility of the capillary viscometer make it ideal for routine viscosity measurements, while the PPC viscometer's shear-controlled flow and broad measurement capabilities make it valuable for more in-depth rheological studies.
- Finally, the study results allow researchers and professionals to select the most appropriate tool for their specific needs. Both viscometers play crucial roles in characterizing fluid properties and optimizing processes in various industries and research fields.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest, and the corresponding author confirms.

AUTHORS CONTRIBUTION

Data analysis, writing: A. Megalingam; guidance and alignment: A.H. Ahmad and N.A. Alang, N. Muhammad and K.Muduli.

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