Development of rheometer for semi-solid high-melting point alloys

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Abstract: A rheometer for semi-solid high-melting point alloys was developed based on the principle of a double-bucket rheometer, with which the solidifying of semi-solid high-melting point alloy melt could be effectively controlled by the control of temperature and the outer force-field; and different microstructures have also been obtained. This rheometer can be used to investigate the rheological behavior under different conditions by changing the Theological parameters. By way of full-duplex communication between the computer and each sensor, automatic control of the test equipment and real- time measurement of rheological parameters were realized. Finally, the influencing factors on torque are also quantitatively analyzed .

Key words: rheometer; rheological behavior; high-melting alloys; semi-solid;

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1 Introduction

The rheological behavior of semi-solid alloy melt is the direct influencing factor of the filling capability of the melt and of product quality. So far, many viscometers have been developed for investigating the rheological behavior of semi-solid alloy under different conditions. For example, a parallel plate compression or rotary shearing rheometer is usually employed to test the rheological behavior of the melt under pressure, the capillary tube rheometer is specifically utilized for mould filling and the double-bucket rheometer is the most widely used one for investigating the transient and steady rheological behavior. However, the viscometers currently available have their limits with the study of rheological behavior for iron and steel. The parallel plate rheometer ca n work only over the range of shear rates from 1 to10^2 s^{-1}. Although the shear rate of the quick drop hammer rheometer, as modified by Yurko [1], based on the parallel plate compression rheometer is improved to 10^4 s^{-1}, it and the capillary tube rheometer can only investigate the transient rheological behavior of the melt while mould filling, which is insufficient to evaluate the comprehensive rheological behavior of semi-solid melt. Additionally, double-bucket rheometers with the operating temperature of less than 1 000 °C [2] are mainly used for low-melting metals such as Sn-Pb, aluminum, and magnesium alloys but not for metals such as iron and steel alloy with high melting point[3-4]. The viscometer for slag can work at 1 400 °C, but usually works in the liquid condition with the maximum viscosity less than 1 Pa • s [5-6] which is much less than that for semi-solid slurry which is several Pa • s.

For lack of a rheometer for high- melting metals, few studies of rheological behavior have been focused on semi-solid iron and steel alloy. This is because it is difficult to accurately control the temperature, torque, and rotational speed; and to keep the solidified phase and inner structure of semi-solid melt at a temperature of more than 1 450 °C. Based on the theory of the double-bucket rheometer, an innovative rheometer for semi-solid high-melting alloy has been developed, as shown in Fig.1. This rheometer incorporates modernized measuring technology and computer technology. Some new measuring methods have also been put forward. Moreover, with this new rheometer semi-solid melt with different microstructures can be obtained while preparing the slurry, and the rheological behavior during delivery and mould filling can also be simulated, which constitute the main processes of semi-solid forming.

2 The principle of measurement

The operating principle of the new rheometer for semi-solid high-melting point alloys is that the semi-solid melt is sheared when there is a velocity difference between the outer bucket and the inner one, and there will

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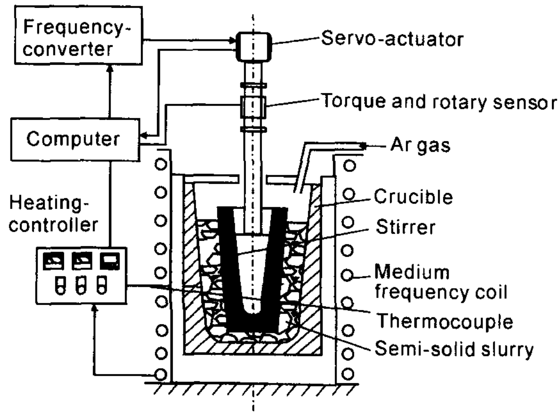


Fig1. Schematic illustration of structure of rheometer for semi-solid high-melting alloy

be a torque on the rotation axis to which the semi-solid melt acts. With automatic control of the shearing parameters and temperature, the solidification of semi-solid melt can be effectively controlled and the semi-solid slurry with different microstructure can also be obtained. By measuring the rotational velocity and torque on the rotation axis, the apparent viscosity of the semi-solid slurry, shear stress and shear rate can be evaluated using equations (1), (2) and (3) respectively, which are deduced from the rotational speed and torque on the inner bucket.

$$\eta_a = \frac{M}{4\pi h\omega}\left(\frac{1}{r^2} - \frac{1}{R^2}\right) \quad (1)$$

$$\tau = \frac{M}{2\cdot\pi r^2 h} \quad (2)$$

$$\dot{\gamma} = \frac{1}{15}\cdot\frac{\pi Rrn}{R^2 - r^2} \quad (3)$$

Where η_a is the apparent viscosity of semi-solid slurry (Pa·s), M is the torque on the inner bucket (N·m), R and r are the radii of the outer bucket and inner one respectively (mm), h is the effective height of the inner bucket immersed in melt (mm) and ω is the angular velocity of the inner bucket (rad $\cdot s^{-1}$). By measuring the rheological parameters the rheological behavior of semi-solid slurry under different work conditions can be evaluated as above.

3 Design of mechanical structure

The rheological behavior of the semi-solid melt is affected not only by the kinetic and mechanical parameters, but also by solidification and phase transformation. Therefore, accurate measurement of the rheological parameters and efficient control of the solidification and phase transformation (especially the shape, size and distribution of the solidified phase) are the key problems for design.

3.1 Design and material selection of inner bucket

The torsional strength of the inner bucket must be high enough to bear big torque which the semi-solid high melting point melt exerts on. The inner and outer buckets, being designed in the shape of hollow taper, not only saves raw material but also prevents the strength of the buckets from decreasing due to non-uniform delivery of the pressure. This is because the polar moment of inertia and the torsion modulus of section of a hollow taper are less than that of solid one, thus the strength and stiffness are improved correspondingly. Also it is easy to release the mould and simplifies the manufacturing process with the taper structure.

The refractory temperature of the inner bucket should also be high enough to work in molten steel. For low-melting alloys, graphite and stainless steel[7] are often chosen as the material for the inner bucket. For high-melting nonferrous alloy, such as bronze, either graphite or Vesuvius 235# ceramic [8-9] is OK. Graphitized and re-crystallized corundum [10-11] can work for high-melting ferrous metal melt, such as iron and steel. However, they can't be widely used because of severe burning loss, brittle fracture at high temperature and high price. A new inner bucket with the raw material of bauxite chamotte and ZrO_2 has been developed. Its refractoriness under a load of 0.4 MPa is higher than 1 600 °C after pressing under high pressure and sintering under high temperature. It can be used extensively as it has high torsional strength, high melting point, low price and a simple manufacturing process. In order to be used over a wide temperature range, the stirrer unit is designed in two parts, with refractory material as the stirrer and heat-resistant steel as the torque transmission part.

3.2 Design of signal collection system

The torque, temperature and rotational speed are three main influencing factors on the apparent viscosity of semi-solid melt. So it is important to collect all signals including temperature, torque and rotational speed simultaneously. As the flow chart of signal collection system in Fig.2, all signals are collected by the A/D data collection card with four serial Com ports. The signals at several millivolts are very weak and the distinguishing ability of the A/D data collection card is ± 5 V, so they are amplified before being collected. In order to reduce SNR (signal-to-noise ratio) and improve the accuracy of

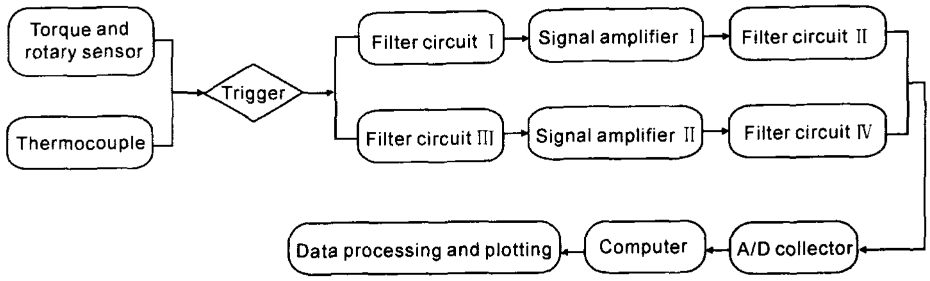


Fig. 2 Flow chart of signal collection system

measurement, all signals are filtered before being fed to the signal amplifier and filtered once again before entering the data collection card. Finally, they are transferred, stored and plotted on the computer.

3.3 Selection of torque sensor and rotary transducer

As seen from equation (1) rotational speed and torque are two basic parameters in the measurement of apparent viscosity. In order to cater for rheological behavior measurement of all kinds of alloys under different experimental conditions, the design should satisfy the following requirements:

(1) The range of rotational speed is wide enough to meet both low shear rate at the moment of delivery of slurry and high rate while quick mould filling. For this rheometer the range is 0-2 900 r/min and the inaccuracy is of 60 pulses per revolution without cumulative error.

(2) Continuously-adjustable and fast mutant shear rate. With a step-less servo-actuator controlled by a frequency-converter, the rotational speed can be controlled continuously according to a set curve.

(3) The acceleration and braking of the step-less servo-actuator can be finished in the short time of about 0.01 s and the time should be adjustable.

(4) A wide range of torque to meet with different solid fraction. The range of torque of this rheometer is 0-500 N ·m and the inaccuracy is 0.2%.

3.4 Design of temperature control system

Temperature is closely related to the solidification and phase transformation of semi-solid slurry. Microstructural criteria (such as solid fraction, size and shape of solid grain) is the intrinsic factor, which affects the apparent viscosity of semi-solid slurry. Therefore, the direct and continuous measurement of temperature and the achievement of a uniform temperature distribution during measurement of apparent viscosity are two key problems for the temperature control.

3.4.1 Method of direct and continuous temperature measurement of iron and steel melt

The conventional temperature measurement method using a thermocouple has many difficulties for high-melting melt such as iron and steel. Firstly, the thermocouple needle will melt or weld with the melt at temperatures above 1 500 °C . The measuring error of quick-thermocouple is high, about 5-10 °C , and also it cannot work continuously. Secondly, there is not enough space for the thermocouple protection tube to be installed in the ring space of 2-4 mm in radius and the response time is long about 1-2 s. Finally, the non-uniform temperature has great effects on the accuracy of a mid-conductor thermocouple and the measured value fluctuates greatly when melting with a medium-frequency induction furnace. The DCTM (Direct and Continuous Temperature Measurementwith bald-node thermocouple)[12] can work continuously at a temperature of 1 700 °C, and has no limitations of space and the surrounding work environment. Its thermal balance error is less than 0.24 °C, the response time is less than 0.3 s and the temperature fluctuation of the melt can be controlled within ±1°C with the help of a PID intelligent temperature-adjusting instrument.

3.4.2 Design of automatic temperature control system

An automatic temperature control system makes the real-time measurement and on-line control achievable. The voltage signal of the thermocouple is acquired at a set frequency and is converted to a digital temperature signal by a volt-temperature program. By comparing the measured value of temperature with the set value, SHIC (algorithm of Simulate Human Intelligent Control) will determine the change tendency (up, down or invariable) of the temperature and will output an appropriate control value E. E will then be transferred to a control voltage of medium-frequency control instrument by D/A converter. The trigger phase angle of SCR (Silicon Controlled Rectifier) changes as the control voltage does and thus the corresponding output power of the medium-frequency furnace is obtained and the temperature fluctuates within a range of less than ± 1°C.

3.4.3 Uniform temperature distribution

There are two ways to get a uniform temperature distribution along the radius. The first is to enlarge the diameter scale of furnace cavity to melt, in other words, to reduce radial temperature difference due to non-uniform magnetic field. The second is to use refractory materials such as asbestos cloth and fused magnesite as an insulating layer around the outer bucket. This can effectively reduce the heat loss of melt along the radius. In fact, the radial temperature difference of the narrow ring space is less than 0.5 °C and is generally ignored.

However, it is harder to get a uniform axial temperature distribution because of non-uniform magnetic field along the axis, which leads to the temperature of both ends of the melt being lower than that in the middle. Also there will be considerable heat loss caused by exposure of the melt to air at the top of furnace. There will be a random additional torque caused by the solidified shell on the surface of melt acting on the inner bucket. Even worse, the solidified shell can hold the inner bucket tightly and break it. So something needs to be done to prevent this. Firstly, the non-uniformity of the magnetic field can be prevented by increasing the height of the medium frequency coil compared to that of the melt. In other words, the height of the crucible should not be greater than 1/3 that of the coil and should be set in the middle along the axis. Secondly, use a temperature control device consisting of a steel heating-board and covering slag. The principle is that the steel heating-board, which is heated by the medium-frequency magnetic field, heats the melt and the covering slag prevents heat from escaping. The magnetic density through the steel plate is less slightly than that through the melt, and there is escape of heat from the top of steel heating board. So the temperature of the steel heating plate is about 1 380 °C , lower than the temperature of the melt by 30-90 °C , but higher than the melting point of the covering slag of 1 350 °C [13]. Therefore, the covering slag is in molten state but the steel plate is not. The thickness of the covering slag in its molten state is about 3 mm and this can reduce the temperature difference along the axis to less than 1 °C by reducing the escape of heat. In addition, it can prevent the melt from oxidizing and can also reduce the additional torque which the covering slag causes on the inner bucket and the random impact torque which covering slag particles cause on the inner bucket [14]. A simplified schematic showing the principle of the temperature control device is shown in Fig.3. The asbestos board and aluminium silicate fiber with good insulation around the steel heating-board can minimize the escape of heat.

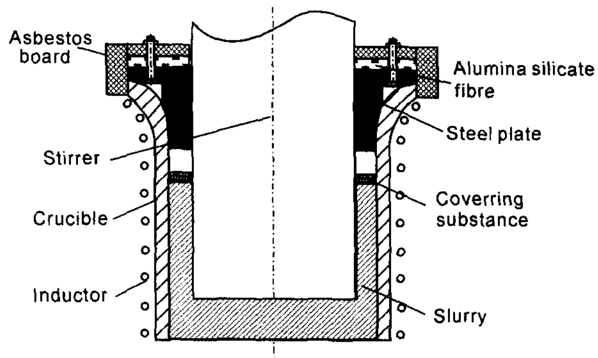


Fig 3. Simplified work schematic of temperature control device

4 Analysis and control of error

The axial temperature gradient, shear heating, coaxiality, end effect and self-friction are the main factors that affect the accuracy of measurement of torque.

4.1 Axial temperature gradient

Axial temperature gradient has a great effect on the microstructure, especially on the solid fraction of the melt. The apparent viscosity of the melt varies greatly along the axis causing the difference in microstructure and the measured value of torque to be not truly representative of the melt The temperature-control device as shown in Fig 3 can effectively reduce the axial temperature gradient.

The distance between the covering slag and the steel plate is so little that the air cannot flow. Therefore, heat transfer by convection and conduction can be ignored and radiation is the main way of heat loss from the melt [15]. The rate of heat flow by radiation per unit area can be expressed as follows

$$q_2 = \frac{5.67}{(\frac{1}{\varepsilon_2}+\frac{1}{\varepsilon_3}-1)}[(\frac{T_2}{100})^4-(\frac{T_3}{100})^4] \tag{4}$$

The rate of heat loss by conduction of the melt and the covering slag can be separately calculated using equations (5) and (6).

$$q = \frac{T - T_1}{\frac{\delta_1}{\lambda_1}} \tag{5}$$

$$q_1 = \frac{T_1 - T_2}{\frac{\delta_2}{\lambda_2}} \tag{6}$$

Where T, T_1, T_2, and T_3 are the temperatures of the melt, top and bottom surfaces of covering slag, and the bottom surface of the steel heating-board respectively (K); ε_2 and ε_3 are the blackness coefficients of the covering slag and the bottom surface of the steel heating-board; λ_1 and λ_2 are the coefficients of heat conduction of the melt and the covering slag (W/m • K); δ_1 and δ_2, are the thicknesses of the melt which has a temperature gradient and the covering slag (mm).

According to the principle of one-dimensional steady-state heat transfer, the heat flow rate per unit area in series between each heat resistance is equal, in other words, $q=q_1=q_2$. The relationship between the temperature of the top surface of the covering slag T_1 and the temperature of the bottom surface the steel board T_3 can be deduced from equations (4), (5) and (6)

$$a(51T_1-50T)^4+T_1-T=aT_3^4 \qquad (7)$$

Where a is a constant, equal to 2.4921×10^{-12}.

As shown in equation (7), T_1 increases with T_3.That is to say, the surface temperature difference $\Delta T=T-T_1$ of the melt decreases when T_3 rises.

Without the steel heating-board, the measured surface temperature of the covering slag is less than 600 °C, ΔT will be more than 17 °C and the melt which has a temperature gradient is about 10 mm in thickness. With the temperature-control device, the maximum temperature difference between T and T_3 is 90 °C. If T is 1 470 °C and T_3 is 1 380 °C, then T_1, will be 1 462 °C, that is to say, ΔT is only 1.8 °C . The covering slag is completely in the molten state, the surface temperature of which is 1 400 °C, thus the additional torque that causes on the inner bucket can be ignored. In all, the temperature-control device consisting of a heating-board and covering slag can reduce or eliminate the error caused by axial temperature difference.

4.2 Shear heating

Shear heating is heat conserved in the melt while sheared and its result is that the temperature of the melt rises and the viscosity decreases correspondingly. The temperature rise due to shear heating is given in equation (8):

$$\Delta T=\frac{M\dot{\gamma}\Delta^2}{2\pi r^2 h\times 8\lambda} \qquad (8)$$

Where, ΔT is the temperature rise caused by shear heating (K), Δ is the clearance between the inner and outer buckets (mm), and λ is the coefficient of thermal conductivity (w/m • K).

As shown in equation (8), the shear heating can be reduced either by enlarging the radius of the inner bucket or reducing the clearance Δ. For this rheometer the temperature rise caused by shear heating can be ignored because the temperature rise is only 0.316 4 K while the torque is 500 N·m giving a shear rate of 300 s^{-1}.

4.3 Coaxiality

The basic principle of a rheometer for semi-solid high-melting alloy is based on the hypothesis that the inner bucket and outer one rotate coaxially. In fact, there is always an offsetting distance because of errors in processing and assembling the parts. Therefore, there will be an additional torque on the axis of rotation. The relationship between this additional torque and the offsetting distance is as expressed in equation (9)[16].

$$\Delta M=\frac{e^2}{e^2+2(R-r)^2}M_T \qquad (9)$$

Where ΔM is the additional torque caused by coaxiality error (N • m), M_T is the measured value of torque (N • m), and e is the offsetting distance (mm).

As equation (9) shows, the additional torque is always bigger than zero, meaning that the measured value of the viscosity of the semi-solid melt is bigger than the real one.

4.4 End effect

End effect, a kind of viscous effect that the melt causes on the end face of the inner bucket, will make the measured value of viscosity bigger than the real one. Experiments prove that the additional torque due to end effect is invariant when the distance between the two end faces is longer than 15 mm and the viscosity of the melt is not less than 0.15 Pa • s. In fact the effect can be reduced, and quantitatively measured by increasing the effective length of the inner bucket immersed in the melt and the axial distance between the inner bucket and the outer one. In theory the influence quantity of the end effect is equivalent to Δh, which is the incensement of the effective length of inner bucket immersed in the melt.

With the distance between the two end faces, the radius and effective length of the inner bucket and the clearance Δ invariable; and the measured values of torque being M and M'when the inner bucket rotates with the speeds of ω and ω'; Δh can be calculated according to equation (10).

$$\eta=\frac{M}{4\pi(h+\Delta h)\omega}\left(\frac{1}{R^2}-\frac{1}{r^2}\right)=\frac{M'}{4\pi(h'+\Delta h)\omega'}\left(\frac{1}{R^2}-\frac{1}{r^2}\right) \qquad (10)$$

In brief,

$$\Delta h=\frac{M'h-Mh'}{M-M'} \qquad (11)$$

For a certain melt and the same device parameters, Δh is constant and can be eliminated.

The self-friction torque ΔM can be directly measured under the condition of no-load. In general, the total error in measurement caused by coaxiality, end effect, shear heating and self-friction can be calculated according the following equation (12).

$$\Delta = \frac{\Delta h}{\Delta h + h}[M_T - \frac{e^2}{e^2 + 2(R-r)^2} M_T - \Delta M] \qquad (12)$$

It's shown by both theoretical calculation and experiments that the error in measurement of apparent viscosity is less than 2% as a whole.

5. System of testing control

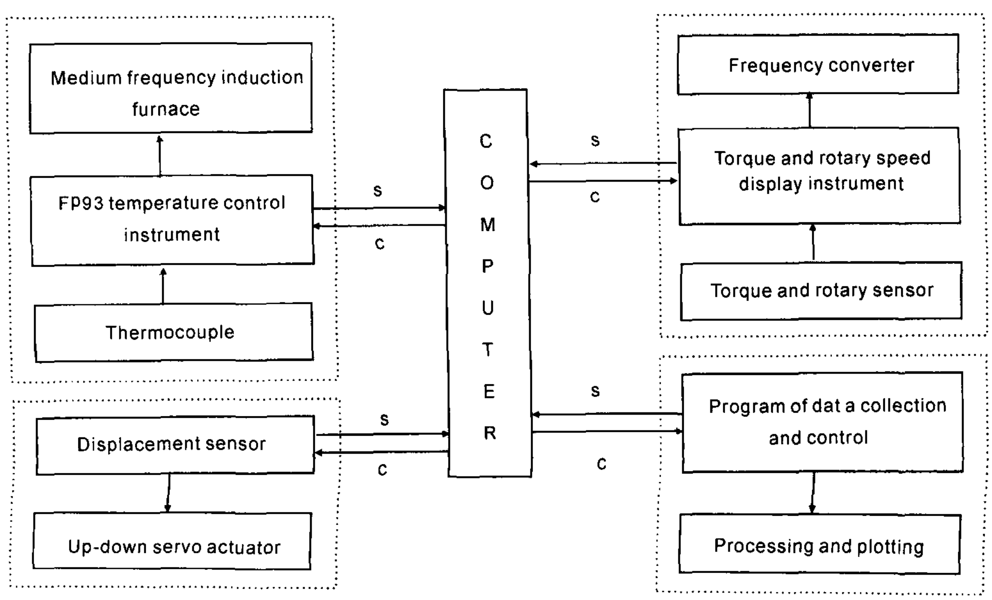
The semi-solid melt has its thixotropy, that's to say, the apparent viscosity is closely related to time. In addition temperature, torque, and rotational speed are all closely related to the rheological behavior of the semi-solid melt. Therefore it is very important to measure all these parameters at the same time.

In order to evaluate the rheological behavior during the whole process of the semi-solid melt preparation, delivery and mould filling, there is a need to change some parameters quickly in order to counter other changes. But it's hard to do this by manual control and the response time is always long. An automatic control system is therefore desirable. The principle is that by way of full-duplex communication, the signals are transferred by RXD (wire for data receiving) from sensor to computer and the control commands are transferred by TXD (wire for data transferring) from computer to each sensor. All the rheological parameters are set using software and the corresponding processes of the experiments are carried out by action executor parts according to orders from the computer. The computer, sensors and action executor parts can communicate with each other as shown in Fig.4. The feedback of signals and the change of parameters can be done within 0.05 s. The control accuracy is so high that the response time on measurement can also be ignored.

6. Conclusions

(1) A new rheometer for investigating the rheology of semi-solid high-melting point alloys has been developed. With it semi-solid melt with different microstructures can be obtained and the rheological behavior can also be studied during the melt preparation, delivery and mould filling; which constitute the main processes of semi-solid formation. In comparison with conventional rheometers, the new rheometer is more precise and reliable, and the rheological parameters can be quickly and accurately measured.

(2) An inner bucket with bauxite chamotte and ZrO_2 has been developed with a softening point under 0.4 MPa of 1 600 °C and this could be used with iron and steel alloys. DCTM, of which the mass measurement error is ±1°C and the response-time constant is less than 0.3 s, has been presented. With the newly-designed temperature control device, the surface temperature reduction of the melt can



S–signal, C–command

Fig.4 Measure and control system of rheometer for semi-solid high melting point alloy be controlled to less than 1.8 °C and some problems such as agglomeration and non-uniform temperature field are also solved.

(3)The main factors that affect the accuracy of measurement of torque (axial temperature gradient, shear heating, coaxiality, end effect and self-friction) have been quantitatively analyzed in this paper. It is shown by both theoretical calculation and experiments that the error in measurement of apparent viscosity is less than 2% as a whole.

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