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분말사출재의 항복응력 측정법 개발

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Development of a Yield Stress Measuring Technique for Powder Injection Molding Feedstocks

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요 약

분말사출재의 항복응력을 효과적이고 간단한 방법으로 측정하기 위해서 수차 장치를 사용한 항복응력 측정장치를 개발하였다. 수차 방식은 벽면 미끄러짐을 고려할 필요가 없는 장점이 있으나 전단률에 따른 점도 변화 측정에는 사용할 수 없는 단점도 있다. 수차 장치의 타당성 검토를 선형유체를 사용하여 검증하였으며, 수차 장치의 양단에서 나타나는 오차도 실험적으로 확인하였다. 분말 현탁액의 일반적인 특징이라고 여겨지는 항복 시 발생하는 순간 최대 토오크는 측정 장치의 제어기 특성에 따른 영향이 큰 것을 확인하고 안정 토오크 영역을 항복응력 계산의 기준으로 결정하였다. 측정되는 토오크에서 수차의 회전 저항에 따른 효과를 제거하기 위해 다양한 속도에서 측정된 토오크를 선형함수로 근사하여 회전 속도가 없을 때의 토오크를 얻었다. 측정 방법의 일반적인 검증을 위해 텅스텐 카바이드 분말과 왁스계 바인더를 이용한 분말사출재의 항복응력을 온도와 분말 충전률 변화에 따라서 측정하였다.

Abstract—In order to measure yield stress of PIM feedstocks simply and effectively, a yield stress measuring technique was developed by a vane method. The vane method had an advantage that there was no wall-slip, while it had a drawback that it could not measure viscosity change at various shear rates. A Newtonian fluid was tested for the appropriateness of the measuring technique. The end effect of a vane was checked to produce an acceptable error. The torque peak has been considered to be developed at yielding of non-Newtonian fluids with yield stress. However, it was influenced very much by control system of the instrument so that the torque value at the stable region was taken to calculate yield stress. Torque at zero rotational speed was obtained by extrapolating the torque values at various speeds to remove the effect of the rotational drag. As general verification, yield stress of feedstocks made of Tungsten carbide powder with wax-based binder was measured at different temperatures and various powder concentrations.

Keywords: Yield stress, Powder Injection Molding, Vane Method, Tungsten carbide powder, wax-based binder, Particulate Suspension

1. Introduction

Powder Injection Molding (PIM) feedstocks can be classified as a highly concentrated particulate suspension. Powder concentration in PIM feedstocks is near to the theoretical limit. It has been known for long that PIM feedstocks have many rheological differences from the polymeric materials. One of the characteristics is existence of yield stress. However, no experimental result about yield stress of PIM feedstocks has been reported. One of the practical applications utilizing yield stress is the development of a realistic rheological model for the CAE analysis. If the size of a part made by the PIM process becomes larger and the tolerance of a part becomes tighter, the CAE analysis would help to produce the part with fewer defects. An accurate prediction by the CAE analysis depends upon the accuracy of input data as well as the numerical technique.

Although yield stress of a solid material has long been known as a typical property, yield stress of a fluid has been a subject for a debate [1, 2]. Hartnett and Hu demonstrated the existence of yield stress by a falling ball experiment [3]. In addition, Astarita proposed yield stress as an engineering reality by explaining the incompressibility of water [4]. In the microscopic sense of view, yield stress can be understood as a transitional phenomenon when a material undergoes structural change during deformation. In the macroscopic sense of view, it can be regarded as a parameter delimiting different deformation behaviors. Anyway, the existence of yield stress is generally accepted now.

Measurement of yield stress of a fluid has also been an engineering challenge. Probably, the earliest definition of yield stress of a fluid might be made for Bingham fluid. In the definition, yield stress was not strictly measured, but estimated from shear stress measured at different shear rates. Some researchers have still practiced the same definition to determine yield stress [5, 6]. However, the other researchers have tried to measure yield stress of a fluid with a concept similar to a simple tension test of a solid material. One of the modes tried in the test is a constant-shear-rate mode and the other one a constant-shear-stress mode. In addition, various types of rheometers have been utilized such as vane, concentric-cylinders, parallel-discs, cone-and-plate, etc. Measuring mode and the type of rheometer should be selected depending upon a material and a measuring instrument.

Among many rheometers, a vane rheometer has been widely accepted for the direct measurement of yield stress with the simplicity and accuracy. Yoshimura and Prud'-homme measured the yield stress of an oil-in-water emulsion by a concentric-cylinders, a parallel-disks and a vane rheometers [7]. They concluded that the vane rheometer produced the most accurate result. They chose a constant-shear-stress mode in the experiment. The concept of the mode is intrinsic in the nature of yield stress. Continuous deformation would occur only when the level of applied shear stress is over yield stress.

Dzuy and Boger measured the yield stress of the Bauxite reside suspension (Red Mud) [8, 9]. They used a constant-shear-rate mode. The concept of the mode is similar in nature to a constant-displacement mode in a simple tension test. They detected a torque peak when a material changed its flowing behavior, and regarded the torque peak as the signal of yielding. Similar behavior was reported by Pignon and coworkers who measured the yield stress of a clay suspension with aqueous base in a cone-and-plate rheometer [10]. When a constant shear rate was applied, shear stress increased sharply until it showed a peak value and decreased slowly to an asymptotic value. They concluded that the peak value corresponded to the yield stress of the suspension.

However, researchers working in the field of electrorheology did not share the same idea to determine yield stress [5, 6]. They found that the level of torque peak in a constant-shear-rate mode changed at different level of shear stress and did not appear at low level of shear stress [5]. Instead of using the peak value, they extrapolated the shear stress obtained at various shear rates to determine yield stress [6]. In the field of electrorheology, the yield stress estimated by the extrapolation method was called as the dynamic yield stress even if yield stress did not include dynamic effect in its nature.

Once yield stress of PIM feedstocks was estimated from shear stress obtained at different shear rates in a previous work [11]. In the work, a parallel-discs rheometer was used, and the wall-slip should be corrected. One of the technical problems in the rheological characterization of PIM feedstocks has been an apparent slip phenomenon at the wall of a rheometer [11, 12]. Although a large number of experiments were performed to correct the wall-slip, the result included substantial amount of error. It was needed that yield stress of PIM feedstocks should be measured in a simpler way. One of the ways to avoid the wall-slip was to use a vane rheometer.

At first, a constant-shear-stress mode was tried to measure yield stress of PIM feedstocks by authors. The result was not reliable due to the characteristics of the instrument used in the experiment [13]. The instrument used in the experiment utilized a well-characterized servo-motor with a rotary encoder. By the nature of the instrument, a constant-shear-stress mode was much faster and more reliable than a constant-shear-rate mode because a constant shear stress could be achieved by controlling the electric current to the amateur of the motor. In the constant-shear-stress mode, angular displacement was detected by the encoder with a finite resolution so that angular displacement finer than the resolution could not be detected easily.

Therefore, a constant-shear-rate mode was tried in the same way as Dzuy and Boger in this study. In a substantial amount of experiments, it was found that the level of torque peak changed upon the level of torque and the rotational speed of a vane. The level of torque peak changed by the rotational speed of a vane could be corrected by extrapolating torque measured at various rotational speeds to the zero speed. However, the torque peak disappeared at a low level of torque. If the torque peak comes from an inherent characteristic of a material at the time of yielding, it should be detected at any torque conditions. Instead of the peak value, estimation of yield stress by extrapolating torque in the stable region produced result that was more reliable. Consequently in this study, yield stress was determined by extrapolating stabilized torque values at different rotational speeds of a vane. In order to check whether the method produced a reasonable result, many tests were made at various temperatures and powder concentrations. In addition, end effect of a vane rheometer was examined experimentally. Through the experimental work in this study, yield stress of PIM feedstocks could be measured in a simpler way without a large amount of experiment.

2. Material and Equipment

2.1. Material

Feedstocks were made of a Tungsten Carbide (W-C) powder with a wax-based binder. The W-C powder was manufactured by Korea Tungsten Co., and was ball-milled in the laboratory. The median of the particle size was 0.8 micrometer and the density of the powder was 12 g/cm^3. The SEM (Scanning Electron Microscope) photograph of the powder is shown in Fig. 1. The wax-based binder had EVA (Ethylene Vinyl Acetate) as a polymer component. The powder concentrations of the W-C feedstocks were 43, 45 and 47 vol. %.

2.2. Equipment

In order to measure the torque developed by a vane in a tank, an instrument of a rotational rheometer manufactured by Physica Co. was used. The instrument could be operated in the shear-rate-controlled or the shear-stress-controlled modes. The maximum measurable torque of the instrument was 50 mNm. The maximum resolution in the torque measurement was one thousandth of the range. However, practically a torque level lower than 5% of the full range was not reliable so that it was desirable to avoid too small level of torque. It was also desirable to avoid a rotational speed lower than 1 rpm (rotations per minute) for reliable measurement although the lowest rotational speed of the instrument was less than 1 rpm. At lower rotational speed than 1 rpm, it was found that the rotational speed was unstable at a certain torque condition.

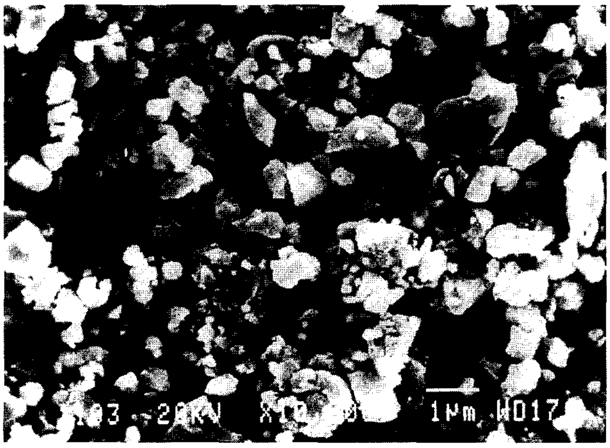

Fig. 1. SEM photograph of the Tungsten carbide powder used in this experiment.

Table 1. Dimensions of the four vanes used in this experiment

Vane / Parameter	Vane-A	Vane-B1	Vane-B2	Bane-B3
Diameter, mm	24	19	19	19
Height, mm	47.1	27.9	37.5	47.1

Originally, a cylindrical bob or a disc was attached to the shaft of the instrument. The instrument measured the torque developed by the bob or the disc. In this experiment, the bob or the disc was replaced with a vane. The vane was immersed in PIM feedstock loaded in a tank. Four vanes were used in the experiment, and all vanes had 4 blades. According to references [9], the number of the blades of a vane does not affect the result of measurement. The dimensions of the vanes are listed in Table 1. The B-series vanes in Table 1 were made to have the same diameter with different lengths. The vanes were used to examine the end effect and development of torque peak. The diameter and height of the tank were 48. 8 mm and 125 mm, respectively.

The dimensions of the vanes were determined by the rule recommended by Dzuy and Boger [9]. The height of a vane should be long enough to reduce the error caused by the upper and bottom surfaces of the vane. The inner diameter of the tank should be at least twice as long as the diameter of the vane. A tank with a large inner diameter could minimize the effect of the stress developed by a vane to the wall of a tank. However, the diameter and the length of a tank were determined within the allowable space of the instrument used in this experiment.

The length of a vane should be determined to ensure high sensitivity of the torque measurement in the instrument. In addition, the torque level should be in a proper range of the instrument. The torque level of Vane-A was estimated from the yield stress value of a PIM feedstock estimated in a previous study [11]. In order to locate the estimated torque level in the middle of the detectable range of the instrument, the diameter and the length were determined.

3. Experiments and Data Processing

3.1. Specimen Preparation

Test specimen was carefully loaded into the tank for a consistent experimental result. The PIM feedstock did not flow easily by its weight due to yield stress and high viscosity. When specimen was loaded into the tank, trapped air bubble should be removed. In order to avoid the thermal loss, a lid made of Bakelite covered the tank. The temperature difference of the specimen in between the upper and lower parts of the tank was within 1~2°C. The standard temperature of a test was set to the temperature at the vertical center of a vane. One of the cautions to be made in the test was that specimen should be pressed and spread by a thin knife before next run. Otherwise, the torque value became too low due to the fracture by yielding of specimen in the previous run. With the same reason, next run started at least 10 minutes after the previous run finished.

3.2. The Shear Stress Calculation

Shear stress was calculated from the torque value with the assumption that shear stress was the same over the entire cylindrical surface of a vane [8, 9]. The torque by the rotation of a vane is the sum of torque developed on the side, top and bottom surfaces of the virtual cylinder. The torque is expressed as below [9].

$$T = (2\pi RH)R\,\tau_w + 2\left[2\pi\int_0^R \tau_e(r)\,r^2\,dr\right] \tag{1}$$

In the equation (1), R and H are the radius and the height of a vane respectively, τ_w is the shear stress developed on the side surface of the cylinder, and τ_e is the shear stress on the top or bottom surfaces. Actually, the shear stress distribution on the top and bottom surfaces could not be obtained without knowing the rheological constitutive equation of the material. Therefore, the shear stress on the top and bottom surfaces was assumed constant and equal to the shear stress of the side surface. This assumption would make error in the shear stress calculation. However, it was reported in a reference that the error was in an acceptable range if the height of a vane was more than twice as big as the diameter [9]. Then, the equation (1) becomes simpler as equation (2). In the equation (2), is the diameter of a vane, and is the shear stress on the side surface.

$$T = \frac{\pi D^3}{2}\left(\frac{H}{D} + \frac{1}{3}\right)\tau_w \tag{2}$$

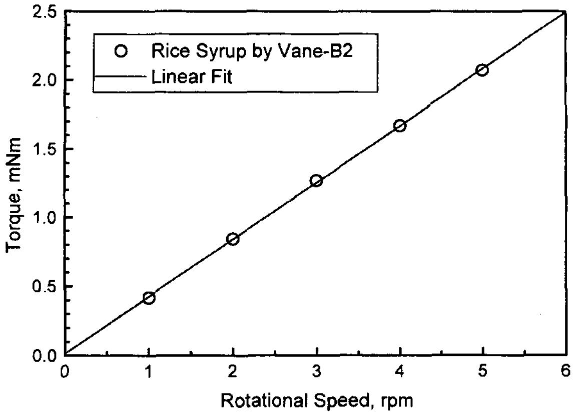

Fig. 2. Torque measured at various rotational speeds for the rice syrup as a Newtonian fluid.

The equation (2) can be arranged in a simpler form to calculate the shear stress from the measure torque value as below.

$$\tau = KT_m$$

$$\text{where } K = \left[\frac{\pi D^3}{2}\left(\frac{H}{D} + \frac{1}{3}\right)\right]^{-1} \tag{3}$$

In the equation (3), T_m is the measured torque value, and K a shape factor determined by the dimensions of a vane.

3.3. Examination of the Vane device with a Newtonian Fluid

Before PIM feedstocks were tested by the vane device, the vane device was examined with a Newtonian fluid. The rice-syrup manufactured by Miwon Ltd. Co. had a range of viscosity detectable by the instrument at a room temperature. It was tested at various rotational speeds by Vane-B2. The results are shown in Fig. 2. The rotational speeds were 1, 2, 3, 4 and 5 rpm, and torque was sampled every one tenth of a second for 5 seconds. As shown in Fig. 2, the torque values were fitted well by a straight line crossing zero point and no distinctive torque peak was detected. The vane device produced an expected result for the Newtonian fluid.

3.4. Torque measurement in the Constant-Shear-Rate Mode

The principle of the method is similar to the simple tension test. With a constant rotational speed of a vane, the torque variation is monitored. The torque level would increase until torque peak occurs and would decrease to a constant level. The constant-shear-rate mode was found more reliable than the constant-shear-stress mode in the

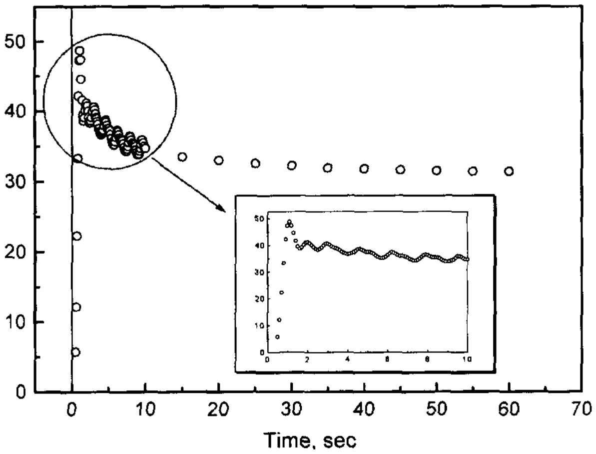

Fig. 3. Torque curve along time for the feedstock with 47 vol.% of powder at 120°C by Vane-A.

preliminary tests. It is said that deformation before the torque peak is caused by elastic property of a material and deformation after the peak is caused by viscous property [10]. This was already examined with a Newtonian fluid in this study. The torque curve of PIM feedstocks showed a peak and slow decrement to a constant level. One of the test results is shown in Fig. 3. The result in the figure was for the W-C feedstock with 47 vol. % of powder at 120°C by Vane-A. The torque peak occurred in a short period so that it was necessary to set the sampling time as fast as the instrument allowed. As shown in Fig. 3, torque was sampled every one tenth of a second during the first 10 seconds of the test. After the first 10 seconds, torque was sampled every 5 seconds up to 60 seconds. The torque peak occurred within 1~2 seconds for PIM feedstocks.

In the blown-up figure in Fig. 3 the fluctuation of torque could be seen. The fluctuation was considered to occur by the control system of the instrument. The instrument used in the experiment utilizes a servomotor as the torque measuring sensor as well as the driver. The electric current to the amateur of the motor is correlated with the torque of the shaft. Rotational speed is detected by a rotary encoder and is fed to the controller. For a constant rotational speed, the electric current to the amateur is controlled with the feedback signal of the encoder. In this type of control system, speed control usually has slower response than torque control. Actually, the rotational speeds recorded in the log file were not always the same as the setting speed, especially in the first few seconds. Therefore, the actual rotational speed at the torque peak should be obtained from the log file. The recorded torque values were corrected by the actual rotational speed.

3.5. Correction of Rotational Speed Effect

When Dzuy and Boger measured the yield stress of the Bauxite reside suspension, the rotational speed was 0.1 rpm [9]. They tested various speeds from 0.1 to 256 rpm to check the effect of rotational speed, and concluded that faster speed than 8 rpm was not recommended [8]. They mentioned that the measured yield stress increased at faster speed than 8 rpm due to flow drag and inertial force. However, the measured yield stress at 0.1 rpm may include the effect of rotational speed even if the speed was low enough. Whether the instrument could detect such a low level of torque at a low speed was another practical problem in the experiment. With a given range of torque in the instrument, the dimensions of a vane should increase for a higher sensitivity. The maximum dimensions of a vane were restricted by the space of the instrument. By the reason, the data obtained at a rotational speed lower than 0.5 rpm were not stable in this experiment. It was found that the torque peak increased as the rotational speed increased. The instrument used in this study could not rotate the vane faster than 5.5 rpm due to the torque limit.

Strictly speaking, even if torque is measured at an extremely low speed, the measured torque includes the effect of rotational speed. Therefore, the effect of rotational speed should be removed by a proper correction. The torque of a shaft in a tangential annular flow is linearly proportional to rotational speed [14]. If the torque measured at various speeds were plotted in a chart along the rotational speed, the data could be fitted well by a straight line, as shown in Fig. 4. If the material were Newtonian fluid, the fitted straight line would intersect

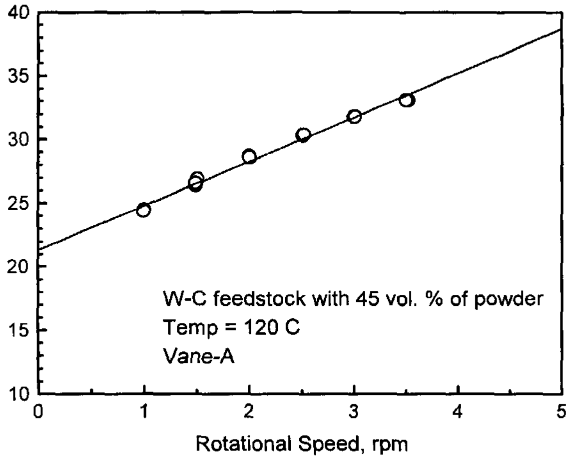

Fig. 4. Torque measured at various rotational speeds and fitted line for the feedstock with 45 vol.% of powder at 120°C by Vane-A.

with the axes of torque at zero. This was already examined with a Newtonian fluid. However, the PIM feedstocks in the experiment produced a non-zero intersecting value with the axes of torque. The torque value at zero speed could be converted to the yield stress without the effect of rotational speed.

3.6. Examination of Torque Peak

In order to determine whether yield stress was calculated from the peak value or the constant level, development of the torque peak was investigated further. Some researchers have been using the constant level to determine yield stress while others have been using the peak value [5-10]. If the occurrence of the torque peak is solely due to a nature of a material with yield stress, the torque peak should be converted to the yield stress of the material. However, if the torque peak could be generated by any other reasons, it would be very difficult to take the torque peak as the standard measure for the yield stress calculation.

It was reported that the torque peak could not be detected at low shear stress level for a material with yield stress [5]. In this experiment, the torque curve fluctuated in most tests and the fluctuation got bigger especially at high level of torque. In order to examine whether the torque peak was made by a nature of material with yield stress, a feedstock was tested by 3 different vanes at a temperature. The 3 vanes are Vane-B1, Vane-B2 and Vane-B3 in Table 1. The vanes have the same diameter with different length. As long as the diameter is the same, the shear stress developed by the vane should be the same at a rotational speed, but the detected torque depends upon the length. The result is shown in Fig. 5. Among the vanes, Vane-B3 with a longest length produced the largest torque peak. The overshoot in the torque curve increased as the torque level increased. From this result, it could be concluded that the fluctuation in the torque curve might be affected very much by the control system of the instrument. Even if the torque peak was still a nature of materials with yield stress, it was very difficult to separate out the effect of the yield stress from the raw data.

Next choice for the yield stress determination was taking the torque value in the stable region. PIM feedstocks showed little difference in the torque curve from the one of Newtonian fluids except the torque peak and diminishing torque level. Fortunately, the torque level did not change much from the highest to the lowest in the torque curve in the case of Vane-B1, as shown in Fig. 5. The torque curve of Vane-B1 was not much affected by the control system of the instrument. The torque level in the stable region could be used to calculate the yield stress with a little amount of error. Therefore, the torque value in the stable region was used to determine the yield stress in this study.

3.7. Examination of the End effect

In the yield stress calculation, the stress distribution on the top and bottom surfaces of the cylinder of a vane was assumed constant. The torque contribution of the top and bottom surfaces was D/3H to the torque of the side surface in the equation. When the ratio of the length to the diameter is 2, the contribution will be 1/6. In order to use the vane device as a reliable instrument for the yield stress measurement, the end effect of a vane should be checked. The 3 vanes with different length and the same diameter were used to examine the end effect. The ro-

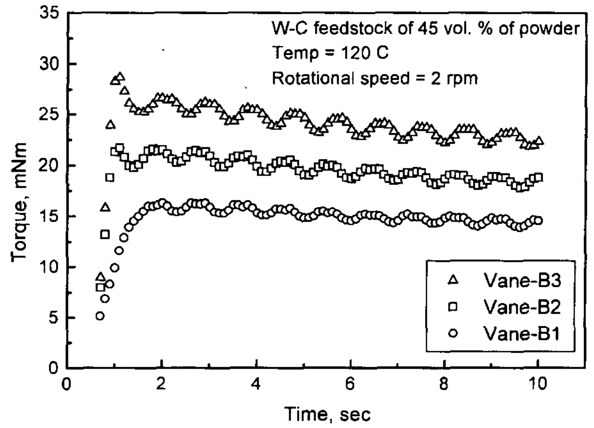

Fig. 5. Torque curve along time for the feedstock with 45 vol.% of pwder at 120°C by various vanes of different lengths at 2 rpm.

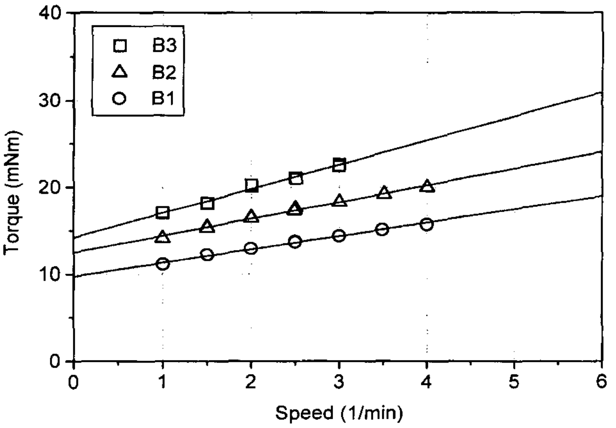

Fig. 6. Torque measured at various rotational speeds by 3 vanes with different lengths and fitted lines.

tational speeds were 1, 2, 3 and 4 rpm, and torque values in the stable region were fitted by a straight line to determine yield stress. The result is shown in Fig 6. The values of the yield stress produced by the 3 vanes were within the experimental error range, about 4 %. The equation (2) turned out to be effective in terms of the end effect of a vane.

3.8. Experimental Conditions

Table 2. Experimental conditions for W-C feedstocks with various powder concentrations and temperatures

Powder vol.% / Temp., °C	47	45	43
130	○	○	×
120	○	○	○
110	×	○	○

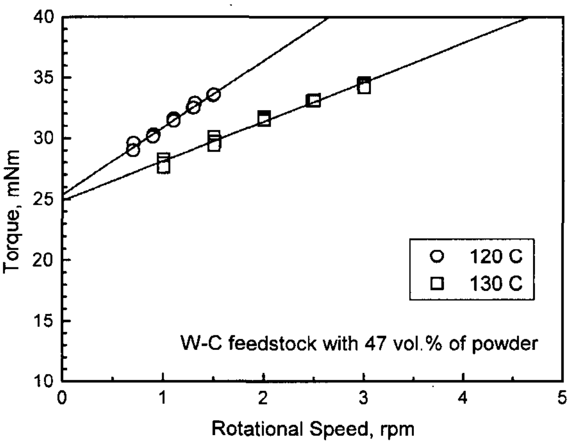

Fig. 7. Torque measured at various rotational speeds by Vane-A for the feedstock with 47 vol.% of powder at 120 and 130°C and fitted lines.

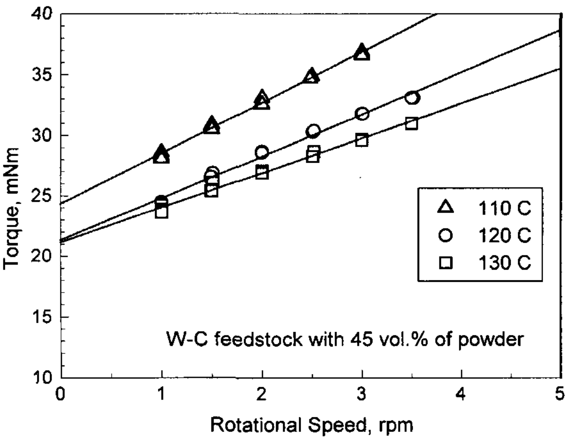

Fig. 8. Torque measured at various rotational speeds by Vane-A for the feedstock with 45 vol.% of powder at 110, 120 and 130°C and fitted lines.

The 3 W-C feedstocks with different powder concentrations were tested at various temperatures. Vane-A was used for the W-C feedstocks. The conditions are listed in Table 2. In the table, the experimental conditions with × mark could not be performed due to the torque limit of the instrument.

4. Results and Discussions

4.1. Effect of Temperature

The torque values of the W-C feedstocks were measured at various rotational speeds and were fitted by a straight line. The results of the W-C feedstocks with 47, 45 and 43 vol. % of powder are shown in Fig. 7, Fig. 8 and Fig. 9, respectively. It can be seen in the figures that the experimental values are well fitted by a straight line. The yield stresses from the torque values at zero rotational speed are plotted along temperature in Fig. 10. As

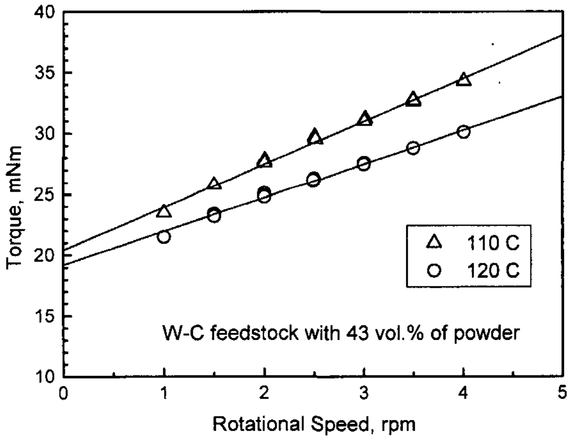

Fig. 9. Torque measured at various rotational speeds by Vane-A for the feedstock with 43 vol.% of powder at 110 and 120°C and fitted lines.

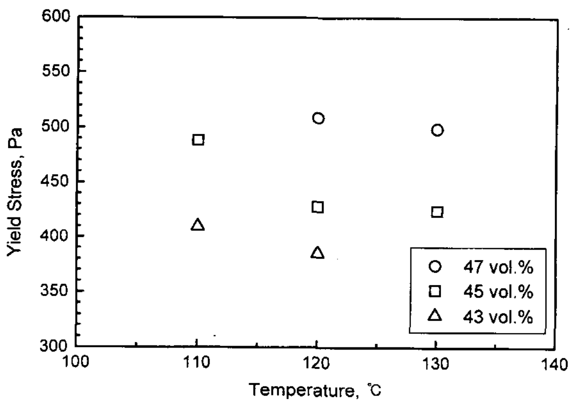

Fig. 10. Yield stress calculated from the torque at zero rotational speed at various temperatures.

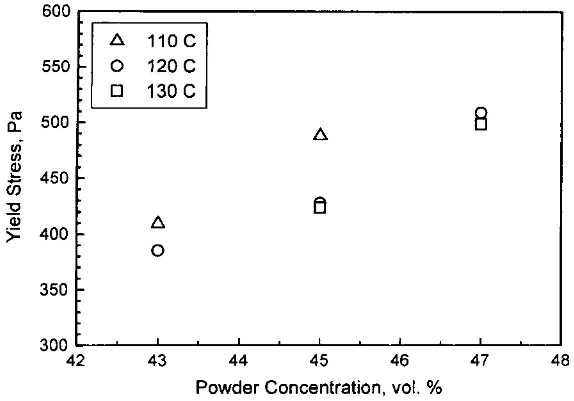

Fig. 11. Yield stress calculated from the torque at zero rotational speed at various concentration.

temperature increased, the yield stress decreased as shown in the figure. The trend could be interpreted by a postulate that the yield stress would be caused by the change of a structure formed by particles, and its magnitude would be related with the strength of the structure. The strength of the structure would depend upon the inter-particular friction. As temperature increased, the inter-particular distance increased due to the thermal expansion of binder. Then, the effective powder concentration of a feedstock would decrease, resulting in the reduced inter-particular friction between the particles. In addition, the binder viscosity would decrease with increasing temperature so that the reduced binder viscosity would weaken the strength of the structure.

4.2. Effect of Powder Concentration

The yield stress was rearranged and plotted along the powder concentration of the feedstocks in Fig. 11. As powder concentration increased, the trend of increasing yield stress was an anticipated result. However, the increasing rate of the yield stress with increasing powder concentration seemed to have a higher slope at a low temperature although the number of experimental data was not enough to draw a conclusion from the result. The trend should be clarified by further research.

5. Conclusions

The yield stress of PIM feedstocks has not been measured before although the existence of yield stress has been known for long. In this study, the yield stress of PIM feedstocks was measured by the vane method. In order to measure yield stress, the constant-shear-rate mode was applied with an instrument that was designed for a rotational rheometer. In order to confirm the rationality of the vane rheometer, a Newtonian fluid, a rice-syrup, was tested. Feedstocks were made of a tungsten carbide powder with a wax-based binder at various powder concentrations. A vane was used to measure the yield stress and 3 vanes were used to examine the development of the torque peak and the end effect of vane. It was concluded that the torque peak could not be used to calculate yield stress because it was affected very much by the control system of the instrument. Instead of the torque peak, the torque value in the stable region was used to calculate yield stress. In order to remove the effect of the drag by the rotation of a vane, the measured torque values at various rotational speeds were fitted by a straight line, and the line was extrapolated to the axes of torque. The intercepted value with the axes of torque by the straight line was converted to the value of yield stress. With the correction method, the yield stress of PIM feedstocks could be measured effectively, regardless of the rotational speed of a vane and the limited range of torque in the instrument.

The W-C feedstocks were tested at various temperatures, resulting in an anticipated trend of decreasing yield stress with increasing temperature. The trend could be interpreted by a postulate that yield stress would be caused by the change of a structure formed by particles, and would be affected by the strength of the structure in the magnitude. In the comparison of the yield stress along the powder concentration, it also showed a reasonable result. However, the experimental results in this study did not fully reveal yield stress affected by many factors because this experimental study was focused on the development of the measuring technique of yield stress. Much experimental work has to be made to understand yield stress of PIM feedstocks. Despite of the limited results in this experiment, it was meaningful that yield stress of PIM feedstocks was directly measured for the first time without the tedious wall-slip correction.

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