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Friction characteristics between metal tool and wood impregnated with phenol formaldehyde (PF) resin during exposure to high pressure

Masako Seki[1] · Soichi Tanaka[2] · Tsunehisa Miki[1] · Ichinori Shigematsu[1] · Kozo Kanayama[2]

Abstract Friction between metal tools and wood under high pressure (≥ 1 MPa) necessarily arises during wood deformation processes and is thus an important factor to be taken into account when wood is processed. We focus on the friction characteristics between a metal tool and wood impregnated with phenol formaldehyde (PF) resin, which is widely used to improve the dimensional stability of wood products. To clarify the effects of the metal tool surface temperature, the PF resin concentration of impregnated solution and the lubricants under exposure to high pressure on the friction characteristics, the friction coefficient (μ) was measured while the wood was compressed in the tangential direction at high pressure (6.7 MPa). The results of μ for higher PF resin concentration differed significantly with the temperature of the metal tool surface. When the impregnated specimens slide under softening temperature (≤ 140 °C), the PF resin squeezing out from the specimen contributed to a decrease of μ because it acts as a lubricant. However, when the impregnated specimens were cured (160 °C), μ increased due to an increase of the sliding resistance on the contact surface. μ was decreased to 0.02 when using a release agent for commodity plastics as a lubricant.

Keywords Friction · High pressure · Phenol formaldehyde resin · Lubricant

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Introduction

Wood forming processes such as compression and bending of solid wood have been researched from various aspects [1]. In these processes, simple shape products can be obtained by wood cell deformation under high pressure (≥ 1 MPa). In addition to these conventional techniques, a new forming process of wood flow forming has been developed to enable the processing of solid wood into more complex forms within a shorter production time [2–4]. This new technique is caused not only by cell deformation, but also by mutual position changes between adjacent wood cells.

After these deformation processes, the deformed wood (products) must have their shape fixed. One effective method to overcome the dimensional instability of the products due to moisture is the impregnation of low molecular weight resins, such as phenol formaldehyde (PF) resin, into the wood prior to the forming process [3–5]. Low molecular weight PF resin can penetrate into the cell walls and softens them, so that PF resin-impregnated woods can be compressed under pressure of more than approximately 2 MPa with heating [6]. After curing under pressure and heat, the impregnated PF resin is polymerized to form a rigid cross-linked network in the cell wall, which prevents recovery of the original shape and results in a compressed and densified form of wood. The PF resin-impregnated compressed wood, which is generically referred to as *Compreg*, has been used for applications such as furniture and flooring because of its high dimensional stability and mechanical properties [7].

When wood is subjected to deformation processes, relatively high pressures (≥ 1 MPa) are generally applied. Under such conditions, the processed wood is exposed to high friction force that arises between the wood and the

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tools used, which can generate wear on the tools and can also have a large impact on the forming limits of the material.

The authors [8–10] have previously reported that the friction characteristics during exposure to relatively high pressure (≥ 1 MPa) are different form those during exposure to relatively low pressures (≤ 0.1 MPa). Under low pressure, the interface contact characteristics have a large influence on the friction characteristics [11]. The friction characteristics under high pressure are not only affected by the interface contact characteristics, but also by the inner structure and mechanical properties of the wood, which vary depending on the pressure, moisture content, and anisotropy of the wood. However, we have not yet reported the friction characteristics of resin-impregnated wood.

Furthermore, the use of lubricants is necessary to achieve successful forming, especially for complex shaped products, although the effect on the friction characteristics during the forming process is still unclear.

The major objective of our study was to clarify the friction characteristics between metal tools and PF resin-impregnated wood during the forming process. The effects of the metal tool surface temperature, the PF resin concentration of impregnated aqueous solution, and lubricants under high pressure on the friction characteristics were investigated. The friction coefficient (μ) between metal tools and PF resin-impregnated wood specimens was measured during compression in the tangential direction at high pressure (6.7 MPa), and the wood specimens and metal tools were then optically inspected to consider the factors that influence the friction characteristics.

Materials and methods

Sample preparations

Wood specimens with dimensions of 30 mm (L; longitudinal direction) $\times$ 30 mm (R; radial direction) $\times$ 5 mm (T; tangential direction) were successively cut from a block of hinoki (*Chamaecyparis obtusa*). The friction surfaces of wood [30 mm (L) $\times$ 30 mm (R)] were finished using an electric planer (Ryobi, HL-6A). The density of a completely dried wood specimen was approximately 0.32 g/cm^3.

Wood specimens were leached in ethanol to remove natural extractives and then completely oven-dried at 105 °C. Dried specimens were impregnated with PF resin having an average molecular weight of less than 500 (PX-341, Aica Kogyo Company) conditioned into various concentrations (solid contents of PF resin were 0, 5, 10, 20, 30, and 50 %) under reduced pressure (0.01 MPa) for 1 h and under pressure (0.8 MPa) for 18 h at room temperature. Impregnated specimens were gently dried under room

temperature conditions for 24 h, air-dried at 35 °C for 120 h, and then vacuum dried completely at 35 °C for 48 h with phosphorus pentoxide (P_2O_5). Weight per gain (WPG) and swelling in the T direction of five specimens due to the treatment were determined using the completely dried weight and dimensions before and after the treatment.

Thermal analysis

A small test piece ($1.5 \times 1.5 \times 1.5$ mm^3) cut from the impregnated and dried wood specimen was subjected to dynamic mechanical analysis (DMA; SII NanoTechnology Inc., TMA SS6100). An oscillating compressive load of 200 ± 100 mN in the T direction was applied to the test specimen and dry nitrogen gas was purged during the measurement, while heating under one of the following two schedules. Firstly, to determine the thermal softening and hardening of wood specimens conditioned with various PF resin concentrations, the test specimen was heated from 60 to 180 °C under a constant heating rate of 5 °C/min. Secondly, to determine the relationship between the heating time and thermal hardening of PF resin-impregnated specimens using PF resin solution at a concentration of 30 %, the test specimen was rapidly heated from 60 °C to each temperature (80, 120, 140, and 160 °C) at a heating rate of approximately 20 °C/min and kept constant at that temperature for 10 min. These analyses were conducted three times.

Measurement of friction coefficient

The PF resin-impregnated and dried specimens were pasted to the upper and under surfaces of a free-cutting brass cube (35 mm) using a cyanoacrylate adhesive (the left hand side of Fig. 1). The specimens were rubbed against a metal tool (SKD11 alloy tool steel) and the friction surfaces of the tools (50 mm $\times$ 100 mm) were finished by polishing. The surface roughness of the sliding direction was measured by the stylus method using a surface roughness tester (Mitutoyo, SJ-500) and surface parameters were calculated based on ISO 4287 [12]. The arithmetical mean deviation R_a, was less than 0.05 μm and the maximum height R_z, was less than 0.69 μm.

Figure 1 (right hand side) shows the setup for measurement of the friction coefficient. The friction coefficients were measured using a dynamic biaxial testing system (Saginomiya, V-1815). The metal tools were fixed to the upper and lower pressure plates, and their surfaces were heated at four temperatures (80, 120, 140, and 160 °C). The friction surfaces of the wood specimens [30 mm (L) $\times$ 30 mm (R)] at room temperature were placed in contact with the heated metal tools without contact of the brass cube with the stem. After 50 s of

Fig. 1 Schematic illustration of the setup for friction coefficient (μ) measurements
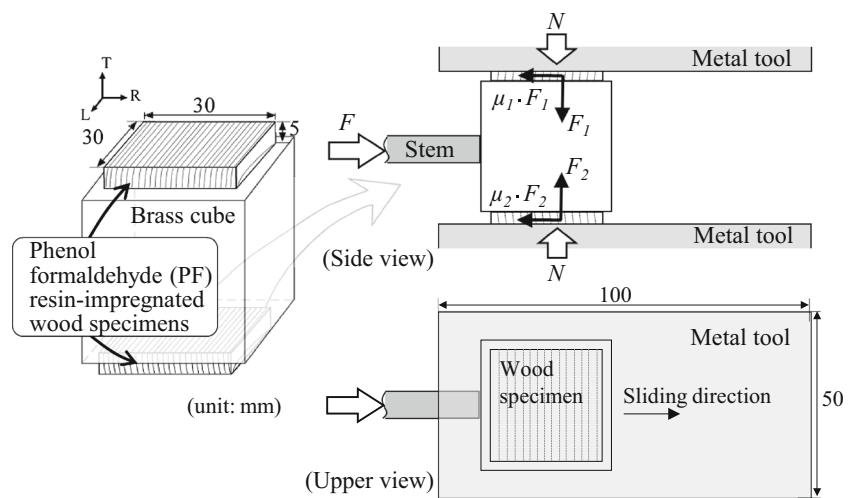
contact with the metal tools, the wood specimens were compressed perpendicular to the contact surfaces at pressing speeds of 10 kN/min up to 6 kN (nominal load N; pressure applied to the nominal contact area (30×30 mm^2) at 6.7 MPa). After the N was loaded to the specimens, 90 s after contact with the metal tools, a stem was moved at a constant speed (30 mm/min) to slide the wood specimen in the R direction with a constant N of 6 kN while the force F, loaded to the stem was measured. Assuming that the force (F_1) and friction coefficient (μ_1) on the upper contact surface were equal to those (F_2 and μ_2) on the lower contact surface, the friction coefficient μ was calculated using the following equation [8–10]:

$$\mu = F/2F_1. \tag{1}$$

The friction surfaces of the metal tools were cleaned with acetone prior to each measurement. In addition to the measurement with the cleaned tool surface, the metal tool surfaces were sprayed with lubricant (Pelicoat S, Chukyo Kasei Kogyo Co., Ltd.), a vegetable oil-based mold release agent for commodity plastics, and measurements were conducted with specimens impregnated with 30 % PF resin that were moved on the metal tool surfaces heated at 140 °C. To investigate the detailed friction characteristics, the sampling time was set as 10 ms. The measurements were conducted twice to confirm the reproducibility of the friction coefficient for each measurement condition.

Results and discussion

WPG and swelling

Figure 2 shows the relation between the PF resin concentration and the average of WPG or swelling in the
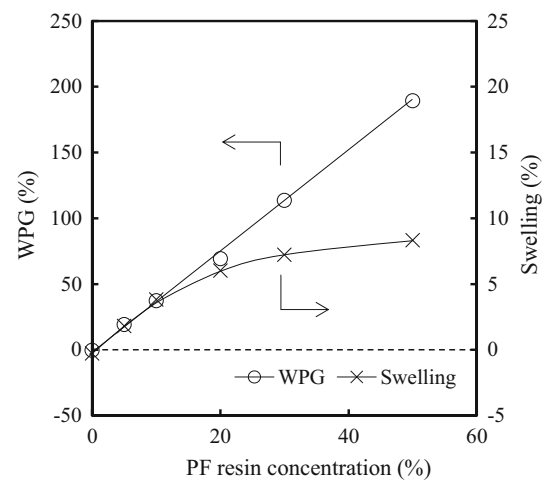

Fig. 2 Relation between the phenol formaldehyde (PF) resin concentration and weight per gain (WPG) or swelling in the tangential direction of the impregnated wood specimens

tangential direction of the impregnated wood specimens. The WPG increased linearly with the PF resin concentration. On the other hand, swelling increased rapidly with the increase in the PF resin concentration at lower PF resin concentrations and then only slightly increased at higher PF resin concentrations (≥ 20 %). It is known that phenolic resin with average molecular weight of approximately 500 can penetrate into cell walls and then swells the woods [13, 14]. From these reports, the results in Fig. 2 suggest that the amount of PF resin in the cell walls increases with the PF resin concentration at lower concentration. In contrast, at higher PF resin concentrations, it is speculated that the amount of PF resin in the cell walls increases only slightly, whereas that in the cell lumens increases more significantly.

Thermal behavior of the wood specimens

Figure 3 shows thermal analysis results with respect to the relative storage elastic modulus. The relative storage elastic modulus ($E'/E'_{60\,°C}$) was defined as the ratio of the storage elastic modulus measured at each temperatures to the storage elastic modulus measured at 60 °C. The relative storage elastic modulus for the untreated sample showed no significant change during the measurement. Impregnation of PF resin into the wood specimens resulted in significant changes in the relative storage elastic moduli, whereby it decreased with elevating temperature from the beginning (60 °C) to approximately 120 °C and then increased from approximately 130 °C. This result indicates that the PF resin-impregnated woods soften at temperatures lower than 120 °C and harden by curing of the PF resin from approximately 130 °C. These changes became more significant with an increase in the WPG of the PF resin-impregnated specimens.

In general, in the wood forming processes, tools used are heated before or after hardening of the wood materials. Therefore, considering the results in Fig. 3, metal tools were heated in the temperature range before and after hardening of the PF resin-impregnated specimens (80, 120, 140, and 160 °C) for μ measurements.

Figure 4 shows the relationship between the normalized storage elastic modulus of the PF resin-impregnated specimens and retention time under each constant temperature. The relative storage elastic modulus showed no significant change at 80 °C for 10 min, a slight increase with the heating time at 120 °C, a gradual increase soon after the measurement start and almost reaching equilibrium after 4 min at 140 °C, and a higher value at 160 °C from the beginning and a rapid increase to reach a maximum value
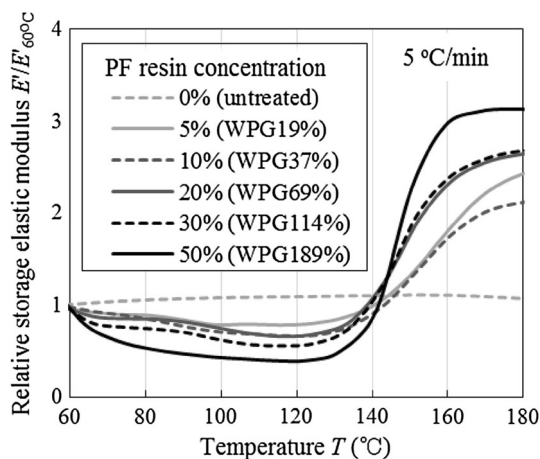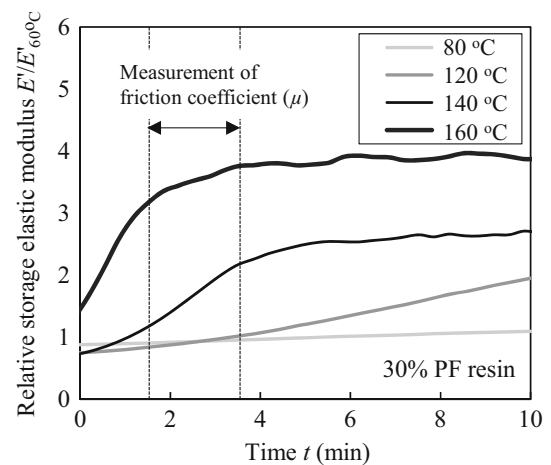

Fig. 4 Relationship between the retention time at each constant temperature and the relative (normalized) storage elastic modulus for phenol formaldehyde (PF) resin-impregnated wood specimens

after 2 min. These results indicate that curing of the PF resin-impregnated specimen proceeds with time at temperatures of 120 °C or higher. Measurements of μ were started after 1.5 min and finished at 3.5 min, during which the degree of curing in the PF resin-impregnated specimens changes with time, especially at 140 °C.

Effect of PF resin concentration and temperature on the friction characteristics

Figure 5 shows the relationship between the stem displacement D, and μ (upper panels) and nominal compressive strain ε (lower panels) for each PF resin concentration and temperature. With $D = 0$ serving as the starting point of the load applied to the stem by the static friction between wood specimens and metal tools. The results indicate similar behavior of μ for PF resin concentrations of 0 and 5 % (upper panels in Fig. 5). At both concentrations, μ increased with the D at 80 and 160 °C, and kept relatively constant value (in some cases vibration is generated by stick–slip) at 120 and 140 °C. On the other hand, the behavior of μ for a PF resin concentration of 30 % was totally different to that for PF resin concentrations of 0 and 5 %, and it differed significantly depending on the temperature. μ decreased with an increase in the D when the specimen was sliding on 80 °C metal tool surface, at which the specimens start to soften. At 120 °C, where the specimen softens, μ oscillated due to stick–slip. μ increased with the D at 140 °C, where the specimens start to harden, and μ became significantly varied during the measurement at 160 °C at which the specimens hardened. The specimens stuck during the measurement at 140 °C and from the start at 160 °C [triangle (∇) in Fig. 5]. The value of μ for a PF resin concentration of 30 % was lower than that for 0 and 5 % at all temperatures.

Fig. 3 Relationship between temperature and normalized storage elastic modulus of the wood specimens conditioned with various phenol formaldehyde (PF) resin concentrations
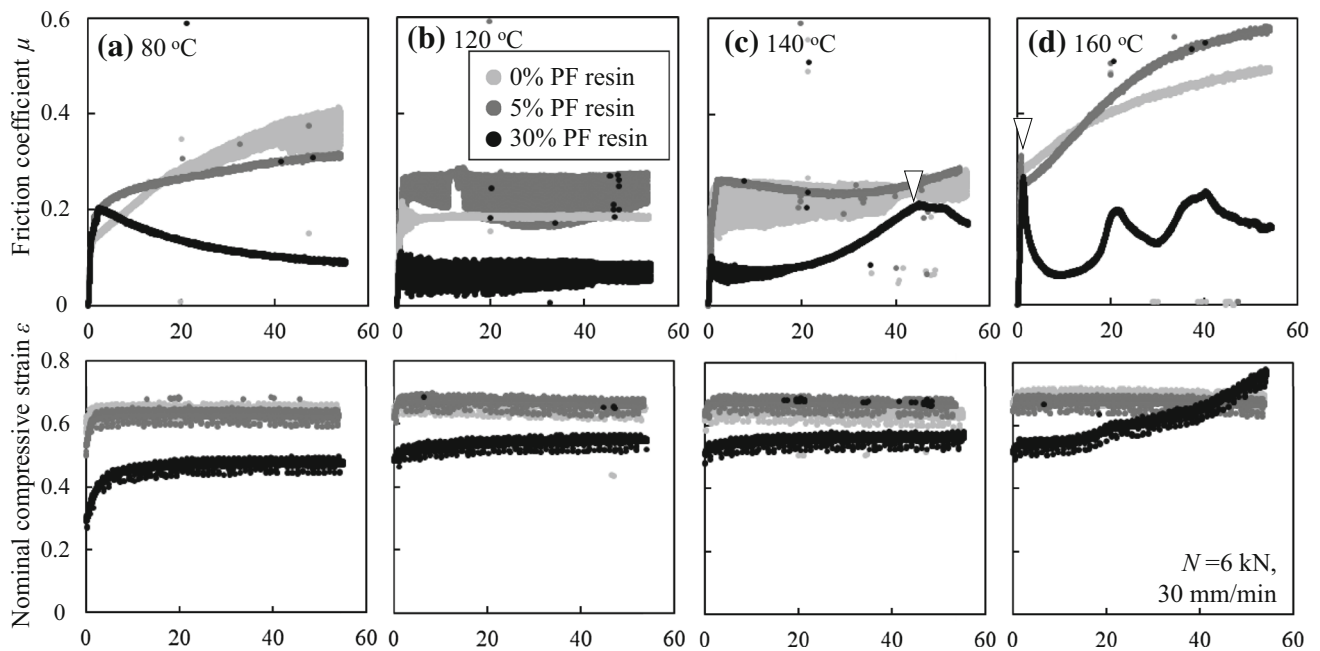
Fig. 5 Relationship between the stem displacement (*D*), and friction coefficient (*μ*, *upper graphs*) and nominal compressive strain (*ε*, *lower graphs*) for each phenol formaldehyde (PF) resin concentration and temperature. *Triangles* show the starting point of the sticking friction of 30 % PF resin

To estimate the deformation of wood specimens during measurement, the nominal compressive strain (ε) was calculated from the magnitude of movement between the upper and lower compression plates of the testing device. The value of ε for PF resin concentrations of 30 % was lower than 0 and 5 % in the range from 80 to 140 °C (Fig. 5a–c). Most of the PF resin in the 5 % PF resin-impregnated specimens was present in the cell walls, while that in the 30 % PF resin-impregnated specimens was present not only in the cell walls but also in the cell lumens (Fig. 2). Therefore, it seems that the 0 and 5 % PF resin-impregnated specimens slide with their cell walls buckled and densified, whereas the 30 % PF resin-impregnated specimens could not be completely compressed because the cell lumens are filled with PF resin. The increase in ε during the measurement at 160 °C (Fig. 5d) was not attributed to compression of the specimens but to the sticking of the specimens to the metal tool surface (as shown in Fig. 6).

Figure 7 shows the appearance of the wood specimens after the μ measurements. The untreated specimens (PF resin concentration of 0 %) showed no significant change at all temperatures examined. In contrast, the appearance of 5 and 30 % PF resin-impregnated specimens showed cured surfaces at 140 and 160 °C ($\triangle$ in Fig. 7). The surfaces of the 30 % PF resin-impregnated specimens were damaged at 140 and 160 °C, which indicate the sticking of the specimens during the measurement. The dotted circles in
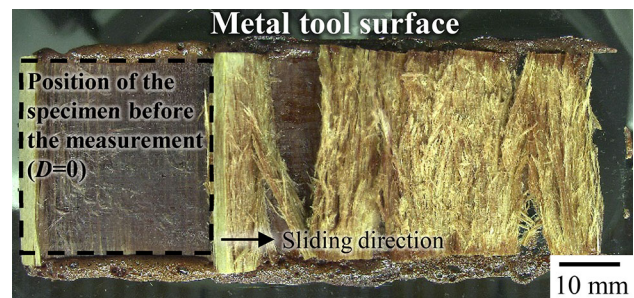

Fig. 6 Optical photograph of the 30 % phenol formaldehyde (PF) resin-impregnated wood specimen stuck onto the metal tool surface after the μ measurement at 160 °C

Fig. 7 indicate the seeping of PF resin from the end grains of the specimens.

Figure 6 shows an optical photograph of the 30 % PF resin-impregnated wood specimen stuck onto the metal tool surface after the μ measurement at 160 °C. The result indicates that when the specimen was moved by stem, shearing fracture was generated inside the specimen where it was not yet hardened, because the resistance on the contact surface was sufficiently large to prevent slip between the metal tool and the wood specimen at 160 °C, at which temperature the specimen was cured. It is known that shearing stress at an interface cannot exceed shearing strength of a material [15]. When the shearing stress at the interface becomes equal to the shearing strength of the

Fig. 7 Appearance of the wood specimens after friction coefficient (μ) measurements. The *triangles* indicate those specimens with cured surfaces. *Dotted circles* indicate end grains of the specimens where phenol formaldehyde (PF) resin is seeping onto the specimen surface
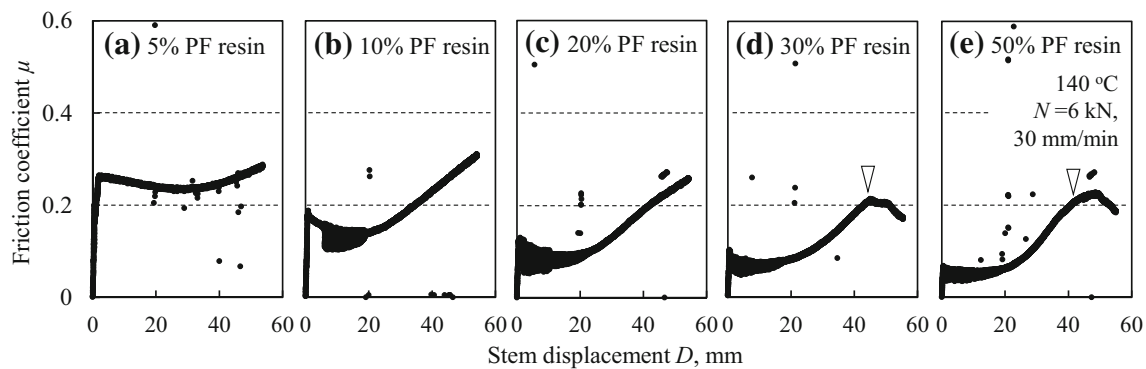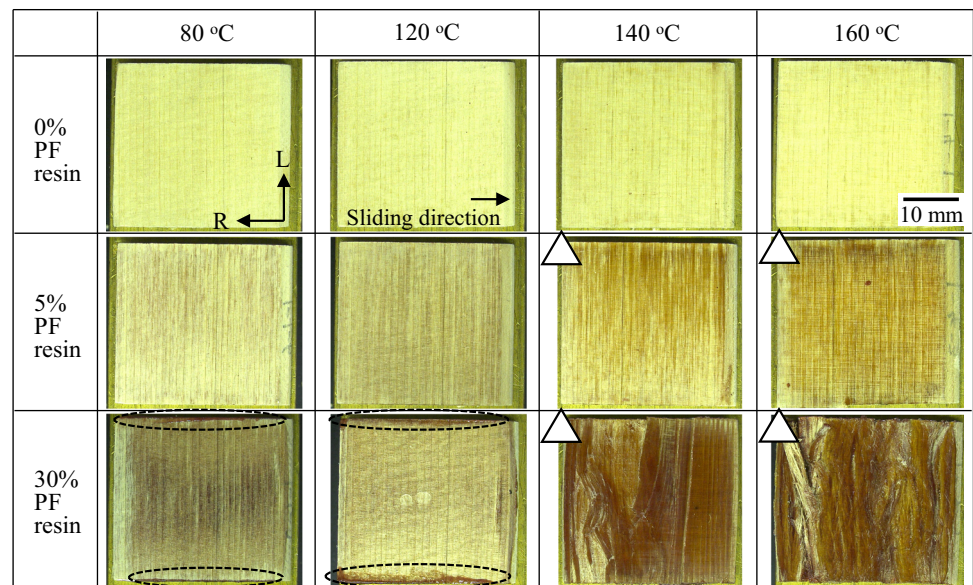

Fig. 8 Relationship between the stem displacement (D) and friction coefficient (μ) for each phenol formaldehyde (PF) resin concentration at 140 °C. *Triangles* indicate the starting point of the sticking friction

material, the slippage at the interface is stopped and shearing fracture is generated inside the material (sticking friction). In this case of sticking friction, the friction force is due not to shearing at the interface, but to shearing fracture [16]. It seems that the variation of μ at 160 °C for 30 % PF resin (Fig. 5) is attributed to repeated shearing fracture of the specimens and rubbing on the fractured surfaces.

The results of Figs. 4 and 5 indicate that the specimen for the 30 % PF resin-impregnated specimen at 160 °C, of which the relative storage elastic modulus was high before the μ measurement [the retention time (t) was up to 1.5 min in Fig. 5], was stuck from the start of the μ measurement (Fig. 4). In addition, the value of μ at 140 °C, at which the relative storage elastic modulus of the specimens increased slightly before the measurement and gradually increased during the measurement ($t = 1.5$–3.5 min in Fig. 4), indicated a significant

increase with the increase in D until it was finally stuck. From these results, it is considered that curing in the PF resin-impregnated specimens near the contact surface increases the sliding resistance on the contact surface and generates sticking friction.

It has been previously reported that μ of water-swollen wood is lower than that of dry wood because the free water acts as a lubricant [10, 17]. Therefore, the lower μ (lower than approximately 0.1) observed for the 30 % PF resin-impregnated specimens at 80, 120, and 140 °C in Fig. 5 seem to be caused by lubrication due to the seeping of PF resin from the specimens (as shown by the dotted circles in Fig. 7).

Figure 8 shows the relationship between the D and μ for each PF resin concentration at 140 °C. The higher PF resin concentrations resulted in lower μ at the beginning of the measurement ($D = 0$–20 mm). From $D = 20$ mm, μ increased with D for all PF resin concentrations.

For the beginning of the measurement, at which point the impregnated specimens are not sufficiently hardened ($t = 1.5$–2.2 min of 140 °C in Fig. 4), it is considered that lubrication by PF resin seepage has a large effect on μ. Figure 9 shows optical micrographs of the metal tool surfaces before and after the μ measurement at 140 °C for 0, 30 and 50 % PF resin-impregnated specimens at each D. Large amount of adherents were seen after the measurement (b)–(e) for 30 and 50 % PF resin specimens, compared to the metal tool surfaces for 0 % PF resin specimen. At the beginning and middle of the measurement ($D = 0$–35 mm, Fig. 9b–d), gel-like adherents ($\triangle$), which seem to be PF resin seeped through the specimen surface, were observed. The amount of adhered PF resin was larger in the 50 % PF resin specimen than in the 30 % PF resin specimen. This suggests that the 50 % PF resin-impregnated specimen has a larger amount of PF resin that acts as a lubricant than the 30 % PF resin-impregnated specimen (Fig. 2), which results in a lower μ for the 50 % PF resin-impregnated specimen due to the higher lubricating effect of PF resin when it is not cured. When D was 35 mm (Fig. 9d), the amount of PF resin adherents decreased, and solid adherents ($\blacktriangle$) were observed when D was 40 mm (Fig. 9e). Thus, the decrease in gel-like PF resin which acts as a lubricant resulted in an increase of μ during the measurement ($D \geq 20$ mm in Fig. 8).

Relationship between PF resin concentration and stick–slip characteristics

Figure 10 shows an enlargement of Fig. 8 for $D = 12$–13 mm. The results indicated stick–slip, which is a phenomenon that involves repetition of the static and sliding states of a specimen [16], for all specimens except the 5 % PF resin specimen. The amplitude and cycle of the stick–slip behavior decreased with an increase in the PF resin concentration. The maximum value of μ during stick–slip which indicates the static friction coefficient [16] also decreased with an increase in the PF resin concentration. It is speculated that smaller static friction coefficient at higher PF resin concentration result from the higher lubricating effect of PF resin seepage through the specimen surface.

Effect of lubricant on the friction characteristics

Figure 11 shows relationship between D and μ for 30 % PF resin-impregnated specimen at 140 °C when the metal tool surfaces were sprayed with a lubricant. The value of μ was approximately 0.02, which was significantly smaller compared with that for no lubricant (Fig. 8d), and there was no significant change during the measurement. It seems that the lubricant sprayed on the metal mold intervened at the interface between the metal tool and wood specimen during the measurement, and thus produced a significantly higher lubricating effect. Figure 12 shows the appearance of the 30 % PF resin-impregnated specimen after the μ measurement with a lubricant at 140 °C. The specimen surface has almost no damage, which indicates the specimen moved only by sliding on the metal tool surface, contrary to the measurements without a lubricant, where the specimen was moved by sliding and sticking (for 30 % PF resin-impregnated specimen at 140 °C in Fig. 7).
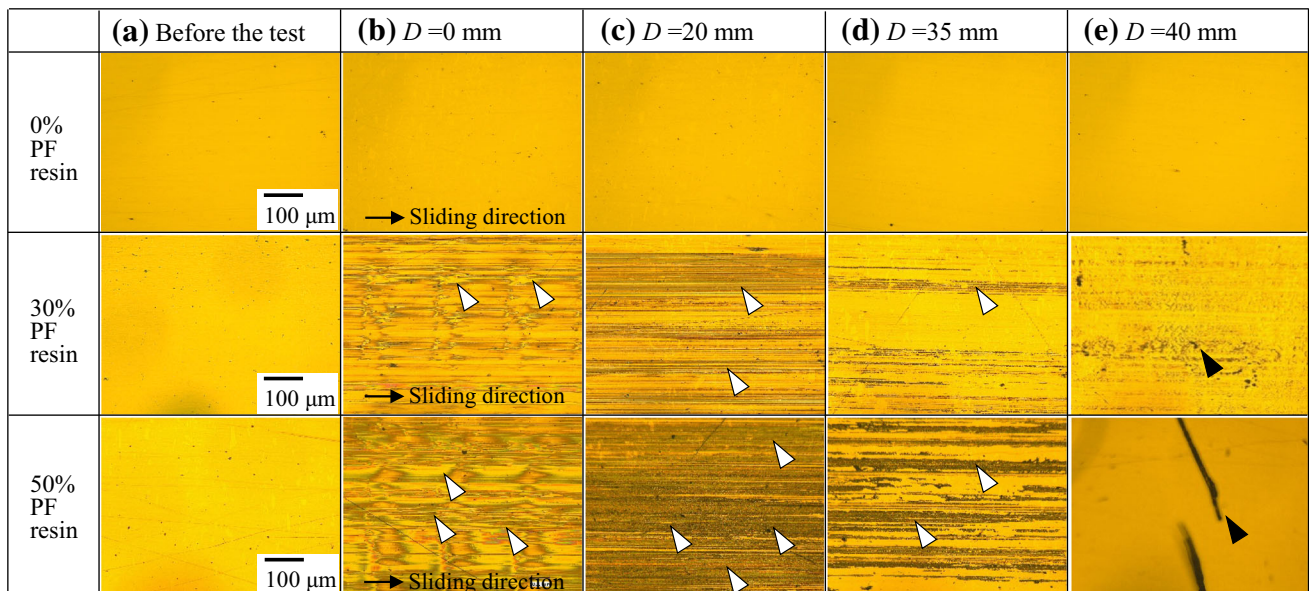

Fig. 9 Optical micrographs of metal tool surface before and after friction coefficient (μ) measurement at 140 °C for phenol formaldehyde (PF) resin concentrations of 0, 30 and 50 % at each stem displacement (D). The *triangles* indicate gel-like adherents
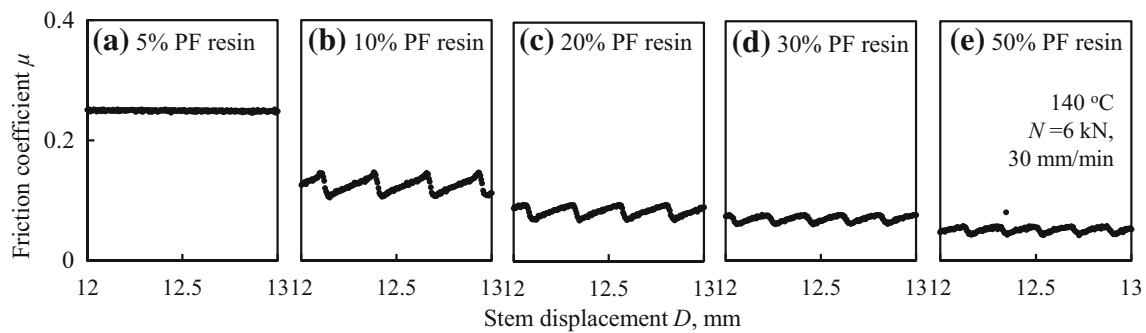
Fig. 10 Relationship between the stem displacement (D) and friction coefficient (μ) for each phenol formaldehyde (PF) resin concentration at 140 °C (enlargement of Fig. 8 of $D = 12$–13 mm)
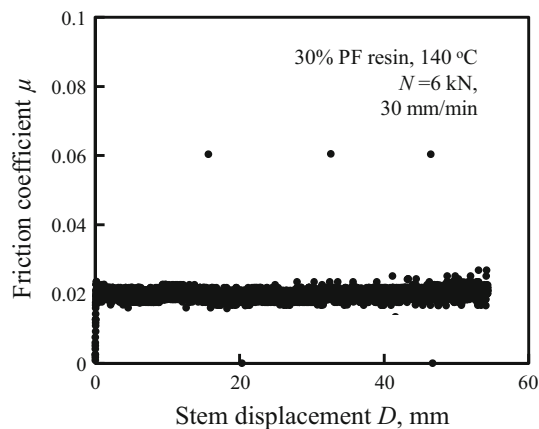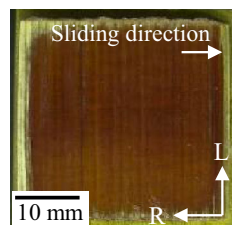

Fig. 11 Relationship between the stem displacement (D) and friction coefficient (μ) when the metal tool surfaces were sprayed with a lubricant (phenol formaldehyde (PF) resin concentration of 30 %, 140 °C)

Fig. 12 Appearance of the 30 % phenol formaldehyde (PF) resin-impregnated wood specimen after the friction coefficient (μ) measurement using a lubricant at 140 °C

Conclusions

The effects of the metal tool surface temperature, the concentration of impregnated PF resin, and lubricants under exposure to high pressure on the friction characteristics between metal tools and PF resin-impregnated woods were investigated. μ was measured while the specimen was compressed in the tangential direction at high pressure (6.7 MPa). The following results were obtained.

1. The change in the behavior of μ for specimens impregnated with the lowest PF resin concentration (5 %) was similar to that for non-impregnated wood. However, μ for a higher PF resin concentration of 30 % differed significantly, depending on the temperature of the metal tool surface. Thus, the softening and hardening of the PF resin-impregnated specimens has a large effect on the friction characteristics. When the specimens slide under softening, PF resin seepage through the specimen surface contributes to a decrease of μ because PF resin acts as a lubricant when it is not cured. However, under hardening, curing in the PF resin-impregnated specimens near the contact surface increases the sliding resistance on the contact surface and generates sticking friction.

2. The amplitude and cycle of the stick–slip and the maximum value of μ during stick–slip which indicates the static friction coefficient decreased with an increase in the PF resin concentration at 140 °C.

3. The use of a release agent for commodity plastics as a lubricant decreased μ to 0.02 and the specimen had almost no damage, in contrast to the specimens with no lubricant, which were damaged by sticking.

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