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ORIGINAL ARTICLE

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Method for measuring viscoelastic properties of wood under high temperature and high pressure steam conditions

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Abstract A method for measuring the viscoelastic properties of wood under high temperature and high pressure steam was developed using a testing machine with a built-in autoclave. A newly developed load cell capable of resisting a steam pressure of $16\,\mathrm{kgf/cm^2}$ and a temperature of 200°C was installed in the autoclave. This load cell could be used to determine precisely the loads while steaming at temperatures from 100°C to 200°C. In addition to load-detection problems, it was necessary to avoid the nonuniform thermal degradation of wood during the measurement process under steaming at high temperatures. This nonuniform degradation could be minimized by shortening the time required for the wood to attain thermal equilibrium using specimens conditioned to the fiber saturation point. According to this method, a stress relaxation curve for sugi (*Cryptomeria japonica* D. Don) wood being compressed while steaming at 180°C was obtained. The stress was seen to decrease rapidly with time, reaching almost zero at 3000s.

Key words Steaming · Viscoelastic properties · Autoclave · Pressure-resistant load cell · Heat-resistant load cell

Introduction

High temperature (or high pressure) steaming is effective as a method for permanently fixing large compressive deformations of wood. Because most of the deformation of compressed wood steamed at temperatures up to 100°C can be recovered by boiling in water, steaming at much higher temperatures is required to obtain permanent fixation. For example, permanent fixation can be achieved by steaming at 180°C for 8min or at 200°C for 1min while the wood is compressed.[1] Considerable permanent fixation can also be achieved by presteaming compressed wood.[2]

To clarify the mechanisms of permanent fixation, detailed information regarding the viscoelastic properties of wood under such conditions is necessary. For this, precise viscoelastic measurement of wood in an autoclave built-in testing machine and the avoidance of nonuniform thermal degradation of wood during measurement are required but have not yet been accomplished. In this paper, a method for measuring the viscoelastic properties of wood under high temperature (or high pressure) steam conditions is described.

Materials and equipment

Equipment

The testing machine with a built-in autoclave is described in Fig. 1. The autoclave, made from stainless steel, can withstand pressures of $30\,\mathrm{kgf/cm^2}$ and temperatures up to 230°C. Two parallel plates to compress the specimen are installed in the autoclave. The lower plate can be moved up and down by an electric hydraulic press placed at the bottom of the apparatus. Displacement of the lower plate is detected by a dial gauge. The load is detected by a load cell on the outside (position A) or inside (position B) of the autoclave. The temperature in the autoclave and of the upper rod near the gasket are measured by thermocouples. The temperature (or pressure) in the autoclave reaches the prescribed level within 10s after steaming.

Wood specimens

To measure the temperature of specimens under high temperature (or high pressure) steam, water-saturated hinoki (*Chamaecyparis obtusa* Endl.) wood specimens 37 (R) × 67 (T) × 80, 40, 20, or 9 (L) mm and water-saturated, air-dried

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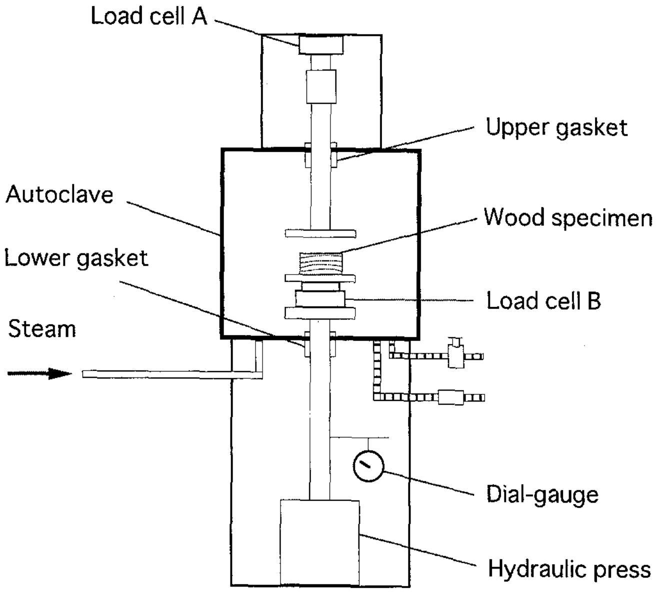


Fig. 1. Testing machine with a built-in autoclave

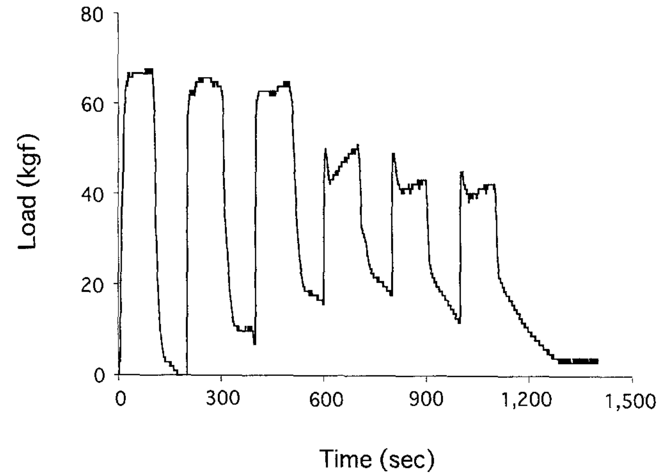


Fig. 2a. Load variation at load cell A in Fig. 1 with time

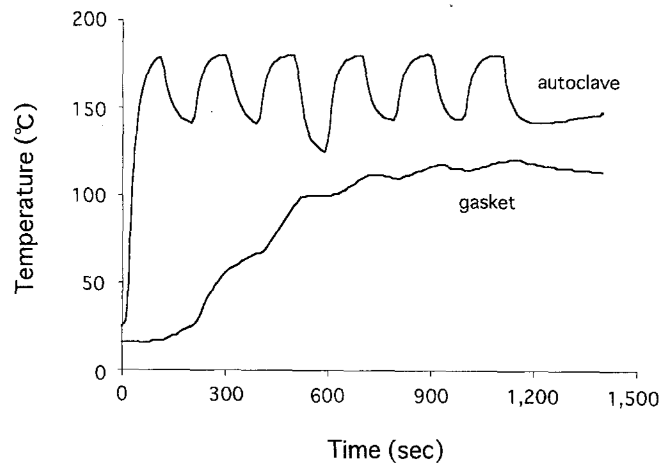


Fig. 2b. Temperature variation near the gasket and in the autoclave over time

hinoki wood specimens 20 × 30 × 20 or 9mm were used. Hinoki wood specimens 20 × 30 × 9mm conditioned to the fiber saturation point (FSP) were also used. To measure stress relaxation, 20 × 30 × 9mm FSP sugi (*Cryptomeria japonica* D. Don) wood specimens were used.

Specimens conditioned to the FSP were prepared as follows: Air-dried specimens were placed over distilled water in a closed chamber for at least 1 week; they were then steamed at 120°C for 30min in an autoclave, cooled slowly in the moisture-saturated condition for about 10h to room temperature, and then placed over distilled water in a closed chamber at room temperature.

Method of load detection

Load measurement by conventional load cell

To maintain the prescribed steam pressure in the autoclave, gaps around rods were sealed with rubber gaskets so the force acting on the upper rod was decreased by friction, and the real force may not be transmitted to the load cell (load cell A in Fig. 1). Changes in load transmitted to the load cell, changes in temperature in both the autoclave and the upper rod near the gasket when the introduction (gauge pressure 10kgf/cm^2) and removal (normal pressure 0kgf/cm^2) of steam were repeated are shown in Figs. 2a and 2b. The load acting on the plate can be obtained from the gauge pressure and cross-sectional area of the rod. As the area of the rod was 7.07cm^2, the load acting on the plate was estimated to be 71kgf. The load transmitted to the load cell approximated the expected value of 71kgf on the first introduction of steam. However, the load gradually decreased with repeated introduction and removal of steam, and it decreased below 50kgf with the fourth introduction of steam. A load of about 20kgf remained following the release of pressure.

The temperature in the autoclave showed a stable variation during repeating introduction and removal of steam, varying between 180°C and 140°C. The temperature of the upper rod near the gasket showed changes different from those in the autoclave. Although the temperature in the autoclave increased rapidly during the first steam introduction, the temperature of the upper rod remained almost unchanged. The temperature of the rod began to rise shortly after the second introduction of steam (from about 200s) and leveled off at about 120°C after about 600s. As load changes of the load cell corresponded precisely to the temperature changes of the rod, the rod was considered to be tightly fastened by the gasket with increasing temperature, and a frictional force of about 20–30kgf occurred. The frictional force acted in a direction that was the reverse of that of the load acting on the rod and corresponded to the difference between the load acting on the plate and that detected by the load cell.

Load measurements by heat- and pressure-resistant load cell

To solve the problem mentioned above, a heat- and pressure-resistant, waterproof load cell (maximum load 500kgf) was developed and installed in the autoclave (load cell B in Fig. 1). That load cell is described in Fig. 3. Strain gauges, sealed at the upper and lower parts using circular rubber gaskets, were coated with a waterproof material to avoid steam penetration. The area of contact between the gaskets and the strain gauges was too small to induce a decrease in the detected load by friction. Furthermore, a leading wire from the strain gauges extending to the outside of the autoclave was covered with a seamless tube made from flexible stainless steel to avoid steam penetration.

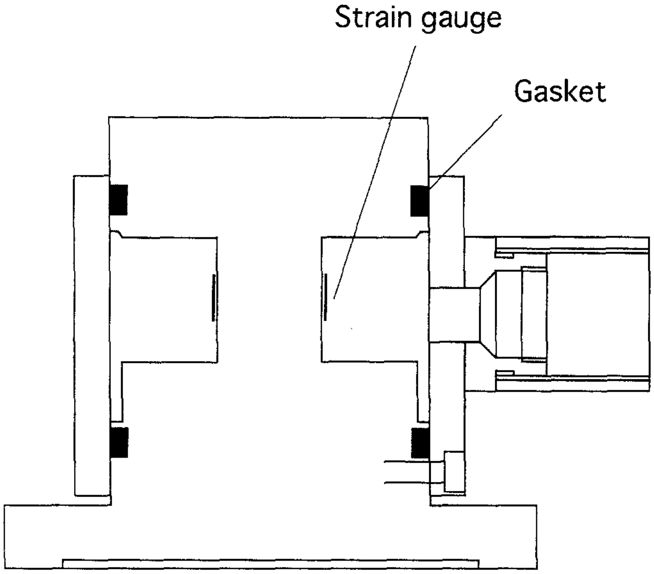


Fig. 3. Load cell capable of resisting a steam pressure of 16kgf/cm^2 (200°C)

To determine whether this load cell operates accurately under such conditions a weight of 7.2kg was applied. The results under steam at 180°C (gauge pressure 10kgf/cm^2) and normal pressure at room temperature are compared in Fig. 4. The measurement under steam was carried out after 30min to minimize baseline drift. The results were almost the same, although the load curve under steam showed slight fluctuations with time. The same results were obtained at temperatures between 100°C and 200°C.

The result at a much larger load (125.6kgf) is shown in Fig. 5. A load of 126kgf was detected at the moment of loading but decreased slightly with time; it had a constant value of 119kgf after 200s. The load decreased to about −9kgf by unloading after 1h. Therefore, the load change by loading and unloading was 127kgf, closely corresponding to the applied load. These results indicated that the newly developed load cell could be used under conditions of high temperature and pressure. The distance between plates due to thermal expansion changed by about 0.1mm but returned to its original value by preheating for 1h.

Uniform heating of wood specimens

When measuring wood properties under high temperature and steam conditions, it is necessary to avoid the nonuniform thermal degradation of wood by rapid heating. Temperature changes at the center of specimens with various sizes (37 × 67 × 80, 40, 20, or 9mm) were measured. Figure 6 shows the results of water-saturated specimens when steamed at 180°C. The temperature at the center of the specimen was measured by a thermocouple inserted through a small hole (1mm in diameter) drilled on the

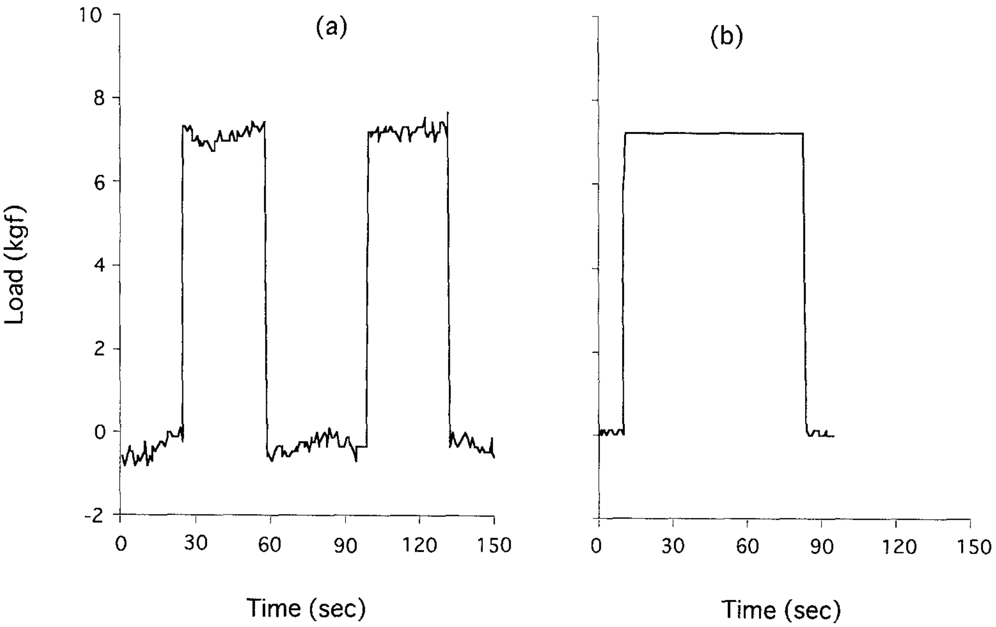
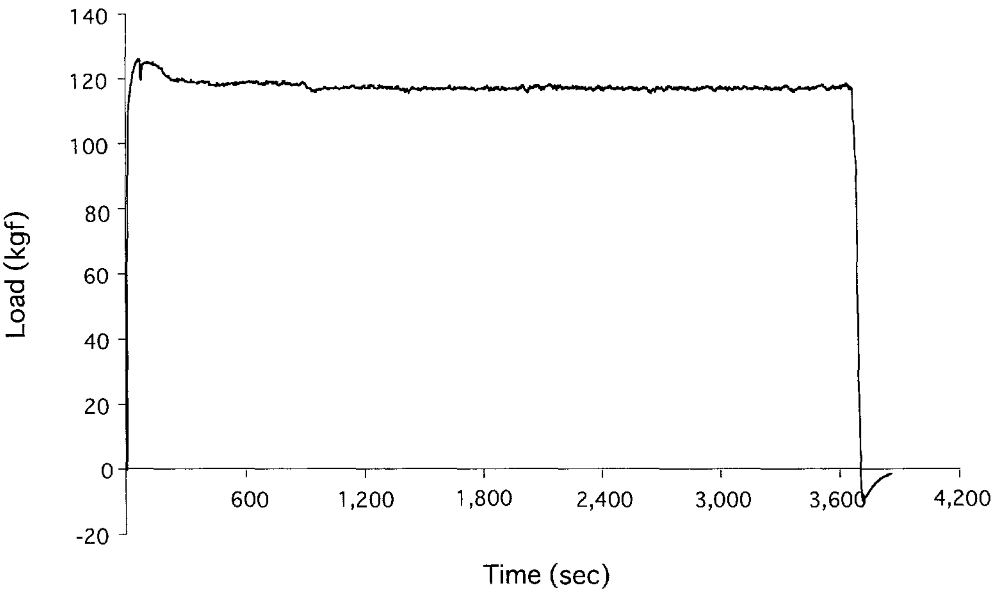


Fig. 4. Load variations under steaming at 180°C (**a**) and at room temperature and atmospheric pressure (**b**)

Fig. 5. Load variation with time while steaming at 180°C



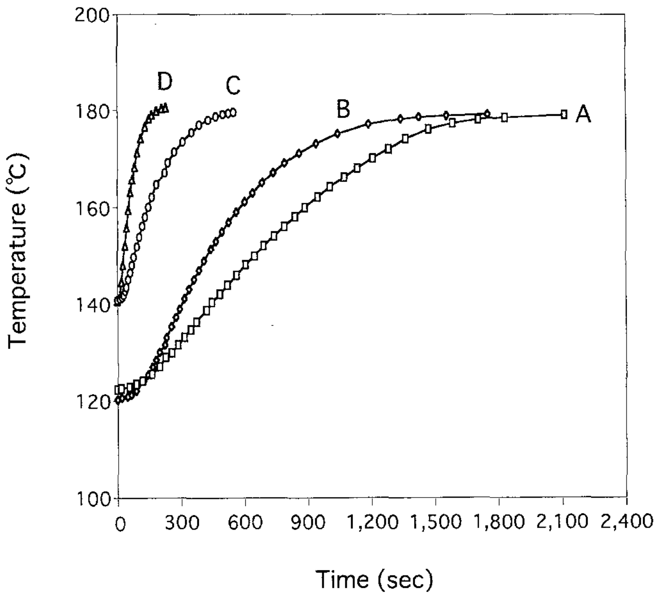


Fig. 6. Temperature variations in water-saturated wood specimens with different dimensions in the longitudinal direction under steaming at 180°C. Dimension in the longitudinal direction: *A*, 80mm; *B*, 40mm; *C*, 20mm; and *D*, 9mm

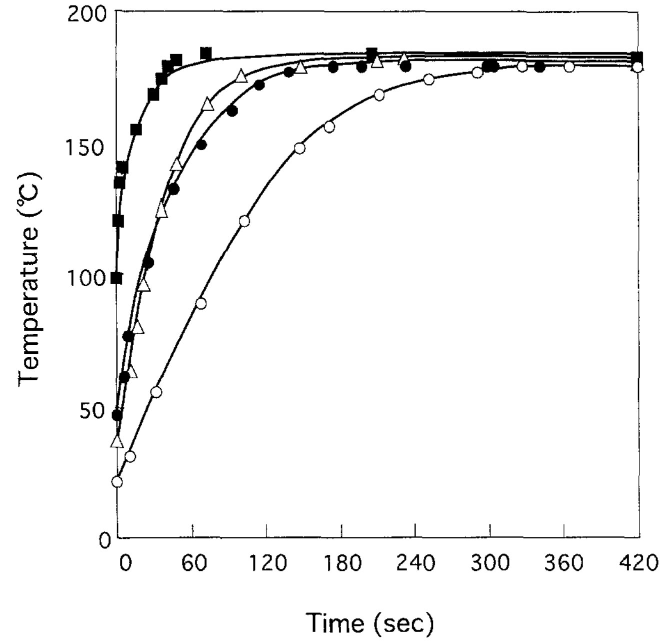


Fig. 7. Temperature variations inside dry and water-saturated wood specimens with different dimensions in the longitudinal direction under steaming at 180°C. *Squares*, dry, 9mm; *filled circles*, dry, 20mm; *triangles*, water-saturated, 9mm; *open circles*, water-saturated, 20mm

tangential plane of the specimen and filled with waterproof adhesive. The temperature of specimens with thicknesses of 80 and 40mm did not reach 180°C even after 1000s. On the other hand, specimens with thicknesses of 20 and 9mm reached 180°C in 500 and 180s, respectively.

Figure 7 shows the results of water-saturated specimens and air-dried specimens under steam at 180°C. The size of the specimen was 20 × 30 × 20 or 9mm. The time required for water-saturated specimens to reach 180°C was about 420s for a thickness of 20mm and 180s for 9mm. The results shown in Figs. 6 and 7 indicate that there was no difference between specimens with different cross-sectional sizes (T-R plane). On the other hand, the temperature of air-dried specimens increased more rapidly than that of water-saturated specimens. The time to reach an equilibrium state for the air-dried specimens 9mm thick was about 60s. The same results were obtained for specimens conditioned to the FSP. These results suggested that when measuring viscoelastic properties of wet wood under high-temperature steam the nonuniform degradation of specimens could be

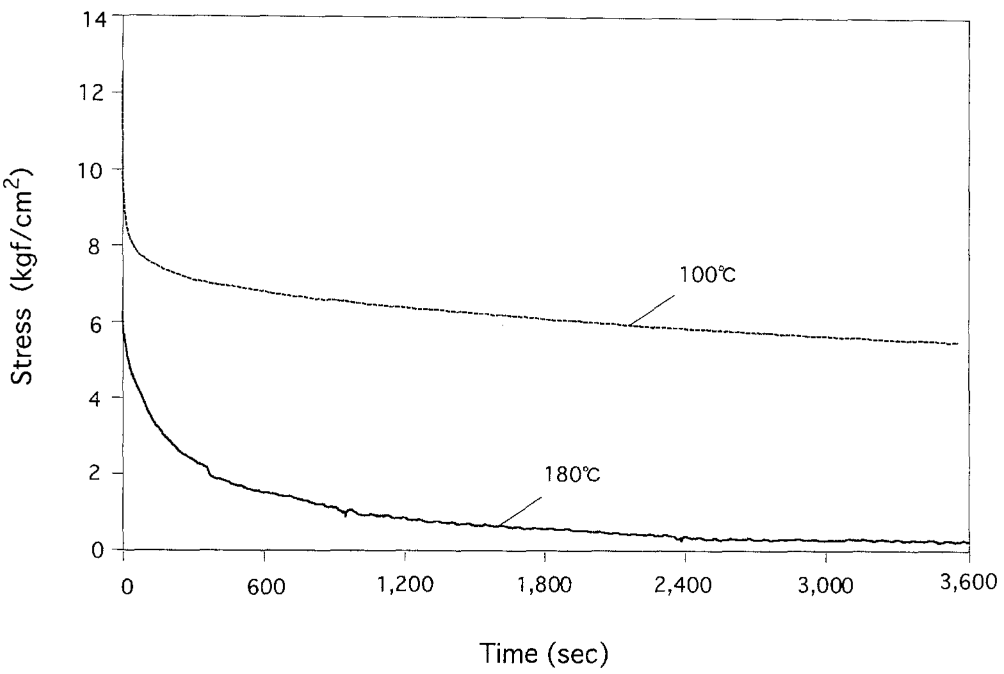


Fig. 8. Stress relaxation curves for sugi wood in compression while steaming at 180°C and 100°C

minimized by shortening the time required for the wood to attain thermal equilibrium using specimens conditioned to the FSP.

Stress relaxation measurement

Figure 8 shows an example of radial compressive stress relaxation curves under steaming at 180°C for sugi wood conditioned to the FSP using the method mentioned above. The measurement was started after the temperature reached 180°C and was held there for 90s with 50% compression. To obtain a large compressive force compared with the force variation due to steam pressure variations in the autoclave, 10 specimens of 20 × 30 × 9mm were compressed simultaneously. The result at 100°C measured in boiled water is also included in Fig. 8. The stress at 100°C decreased instantly and then continued to decline gradually after 300s. The relaxation curve at 180°C was considerably different from that at 100°C. Here the stress continuously decreased and reached 0.3kgf/cm^2 after 40min (2400s). It was thought that the stress decrease at 180°C was due to degradation as well as the molecular movement of the cell wall polymers. The stress did not disappear in 8min at 180°C, although it had been reported that the compressive deformation was perfectly fixed at the same time and temperature conditions.[1] This fact includes an important suggestion that may clarify the mechanism of permanent fixation. It will be described in the next paper.

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