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Shear modulus and thermal properties of gutta percha for root canal filling

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SUMMARY The purpose of this investigation was to investigate the rheological properties of four commercially available gutta perchas for root canal filling. The relaxation modulus [Gr(0): instantaneous shear modulus] and specific volume of their materials were examined. In addition, the quantity of heat was also studied by differential scanning calorimeter. In a lower temperature range than the first-order transition temperature (melting point), the Gr(0) values of each material were almost identical. A marked decrease of Gr(0) was observed at the melting point, and the range of the first-order transition temperature at heating was from 42·0 to 60·0 °C. At higher temperatures than the first-order transition temperature of each material, a considerable difference in Gr(0) values was observed. The transition temperatures obtained by the results of the Gr(0), specific volume and quantity of heat agreed with one another. A marked specific volume change was observed at the first-order transition temperature. The technique using melted gutta percha may not be favourable compared with the conventional lateral condensation technique because melted gutta percha undergoes a large amount of shrinkage during setting.

KEYWORDS: gutta percha, shear modulus, specific volume, quantity of heat

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Introduction

Gutta percha is one of the most popular materials for obturating the root canal space, and a substitute material for gutta percha has not yet been developed. The lateral condensation technique, which fills using one main point and a number of accessory points placed in the root canal space with sealer, has been widely accepted and used clinically for a long time. Additionally, a number of techniques for root canal filling using gutta percha have been developed, and most of them make use of its properties of thermoplasticity and solubility with organic solvent. Especially since the vertical condensation of warmed gutta percha was proposed (1) and the injectable thermoplasticized gutta percha was introduced (2), a variety of techniques using warmed gutta percha have been developed, and many evaluations of the sealing ability of these techniques compared with the lateral condensation technique have been carried out (3–5). However, most of them reported that warmed gutta percha techniques were not necessarily superior to the lateral condensation procedure, and the use of sealer was encouraged frequently (6, 7). At present, warmed gutta percha techniques have not replaced the lateral condensation technique. The lack of sealing ability is due not only to the absence of adhesion between the root canal wall and the gutta perch but also to the particular shrinkage at setting. Studies on the mechanical properties (8, 9) and thermal properties (10, 11) of gutta percha have been reported, but their number is limited and there remain many unclear issues with the thermomechanical properties. Therefore, an investigation of the rheological properties of gutta percha for root canal filling should help to analyse the problem of the utility and sealing ability of warmed gutta percha.

The purpose of this study was to clarify the rheological properties of gutta percha, and to discuss whether

the method using warmed gutta percha is proper for closely obturating the root canal.

Materials and methods

Materials

Four commercial gutta perchas for root canal filling, comprising three injection type and one plastic carrier type, were used. They are listed in Table 1, together with the respective manufacturers and code names.

Measurement of relaxation modulus

The relaxation modulus was measured using a trial apparatus. A schematic representation of the apparatus and the specimen case (S) are shown in Fig. 1. The inside dimensions of this case were 8 mm length, 12 mm width and 10 mm height. Each material was melted and transferred into this case. A thin stainless steel plate (8 mm in width and 0·1 mm in thickness) was inserted 5 mm into the material at the centre of specimen case before setting and was parallel to the wall of specimen case. The specimen case could be moved up and down by turning the handle (H). The shear load produced on the plate surface was measured by a U-type strain gauge (G), and the electric signals from the U-type strain gauge were amplified (A), and recorded (R). The specimen case was surrounded by a heating and cooling box (M, C), and the specimens were heated and cooled at the rate of 0·5 °C min^{-1}. The range of measurement temperature was from 24 to 75 °C. The contact area of material to stainless steel plate was 80 mm^2, and the shear strain (γ) was 0·0125. The measurement time was 6 s, and the relaxation modulus [Gr(t)] was calculated as follows:

$$\mathrm{Gr}(t) = f(t)/\gamma$$

Table 1. Materials used

Material	Manufacturer	Type	Code
Obtura II	Obtura corporation, USA	Injector	OB II
Obturation gutta hard	Toyo chemical, Japan	Injector	OBH
Thermafil	Tulsa dental products, USA	Plastic carrier	TH
Ultrafil regular set	Hygenic corporation, USA	Injector	ULR

where $f(t)$ is the shear stress. The measurement was repeated three times for each material and the central value was employed.

Measurement of specific volume

The specific gravity of each specimen at 24 °C was measured using a pycnometer for calculating specific volume (reciprocal of density). Furthermore, for each specimen, the cubical expansion and shrinkage at heating and cooling were measured using a dilatometer. The cylindrical specimen (7·0 mm in diameter and 15·0 mm in length) was inserted into the sample case of the dilatometer. After excluding air from the dilatometer using a vacuum pump (BSW-50)* for 4 h, the volume around the specimen was filled with mercury. The specimen case of the dilatometer was then soaked in silicone oil, and the specimen was heated and cooled at the rate of 0·5 °C min^{-1}. The temperature range of measurement was from 24 to 75 °C. The height of mercury in capillary tube of the dilatometer was directly read with a microscope. From these values, the specific volume was calculated at each temperature. The measurement was repeated three times for each material and the central value was plotted. In addition, the percentage of specific volume change (λ) at cooling between 75 and 24 °C was calculated using the following equation:

$$\lambda = \frac{S_{75} - S_{24}}{S_{75}} \times 100(\%)$$

where S_{24} and S_{75} are the specific volumes at 24 and 75 °C, respectively.

Measurement of quantity of heat

A differential scanning calorimeter (DSC; DSC-7000)† was employed for this study. The sample was heated and cooled at the rate of 0·5 °C min^{-1}. The range of measurement temperature was from 24 to 75 °C. The peak temperatures that arose at the exothermic or endothermic reaction were obtained. The measurement was repeated three times for each material and the central value was plotted.

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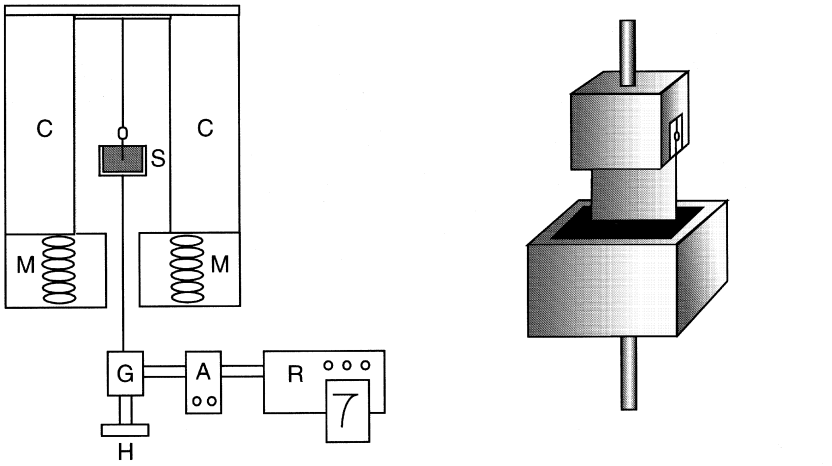

Fig. 1. Schematic representation of appratus used.

Results

Relaxation modulus

The variations of moduli of the four materials are shown in Fig. 2. The relaxation modulus was shown as Gr(0). The Gr(0) values of all materials were 9·6–13·4 MPa at temperatures lower than the first-order transition temperature. On heating, a marked decrease of Gr(0) was observed for all materials; 60·0 °C for Obtura II (OB II), 42·0, 53·0 °C for Obturation gutta hard (OBH), 51·5 °C for Thermafil (TH), and 47·0 °C for Ultrafil regular set (ULR). At higher temperatures than the first-order transition temperature, the highest Gr(0) value was seen on OB II, followed by OBH, TH and ULR in this order. On cooling, marked increases of Gr(0) were observed for all materials; 43·5 °C for OB II, 36·0, 45·0 °C for OBH, 33·5 °C for TH and 31·0 °C for ULR. Except for OBH, the behaviours of Gr(0) were different between heating and cooling, and the setting points were somewhat lower than the melting points. For OBH, the behaviours of Gr(0) at heating and cooling were almost identical.

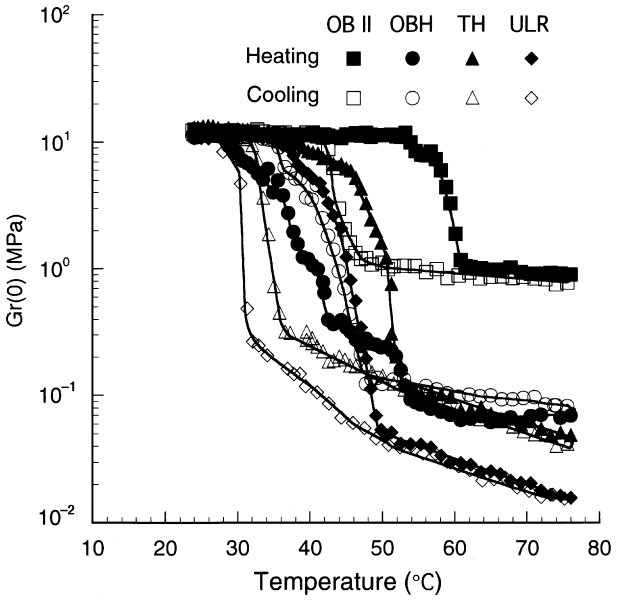

Fig. 2. Variation of Gr(o) with temperature of four materials (heating and cooling rate:0.5°C/min).

Specific volume

The results of specific volume change of the four materials in response to temperature variation are shown in Fig. 3. A marked increase of specific volume was observed between 41·0–56·0 °C on heating and a marked decrease of specific volume was observed 28·0–46·0 °C on cooling. The highest specific volume was TH, followed OB II, ULR and OBH in this order, for comparisons at the same temperature. The percentage specific volume changes (λ) when specimens were cooled from 75 to 24 °C are shown in Table 2. The range of the values was from 4·15 (ULR)–5·05% (OBH).

Quantity of heat

The results of DSC are shown in Fig. 4. On heating, two or three peaks were observed between 40·8 and 62·2 °C for each material. On the contrary, on cooling, only one

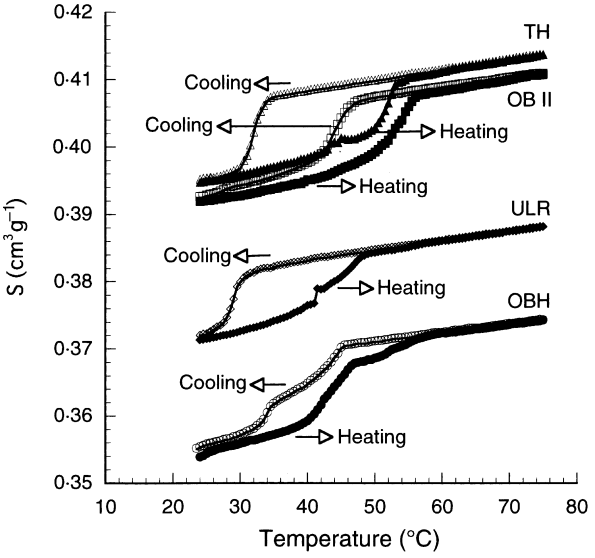

Fig. 3. Variation of specific volume (s) with temperature of four materials (heating and cooling rate:0.5°C/min).

Table 2. The percentage of specific volume change at cooling procedure (75 to 24 °C) and melting point of four materials

Material	λ%, mean (s.d.)	Melting point (°C)
Obtura II	4·48 (0·030)	60·0
Obturation gutta hard	5·05 (0·080)	53·0
Thermafil	4·55 (0·064)	51·5
Ultrafil regular set	4·15 (0·066)	47·0

peak was observed between 29·7 and 43·8 °C for three materials, OB II, TH and ULR. The peak temperatures at cooling were observed to be lower than those on heating. For OBH, two peaks were observed at cooling; one of these was observed at the same temperature as one of the peaks for heating. Transition temperatures obtained from the results of relaxation modulus and dilatometric analysis almost agreed with the peak temperatures obtained from DSC.

Discussion

Gutta percha for root canal filling is a composite material. The main components reported in previous studies (9, 12) were zinc oxide, pure gutta percha (trans-1, 4-polyisoprene), wax or resin as plasticizer and barium sulphate. Although the melting point of pure gutta percha was 80 °C, the depression of the melting point was observed for each material in this study. It was supposed that the cause of this depression was the presence of wax or resin in gutta percha for root canal filling. Furthermore, the shear modulus [Gr(0)] of gutta percha for root canal filling obtained by the measurement of stress relaxation (heating and cooling rates were 0·5 °C min^{-1}) was as follows: (a) although the Gr(0) values of the four materials were almost the same as one another for temperatures lower than the first-order transition temperature, large differences in Gr(0) values of the four materials were observed at temperatures higher than the melting

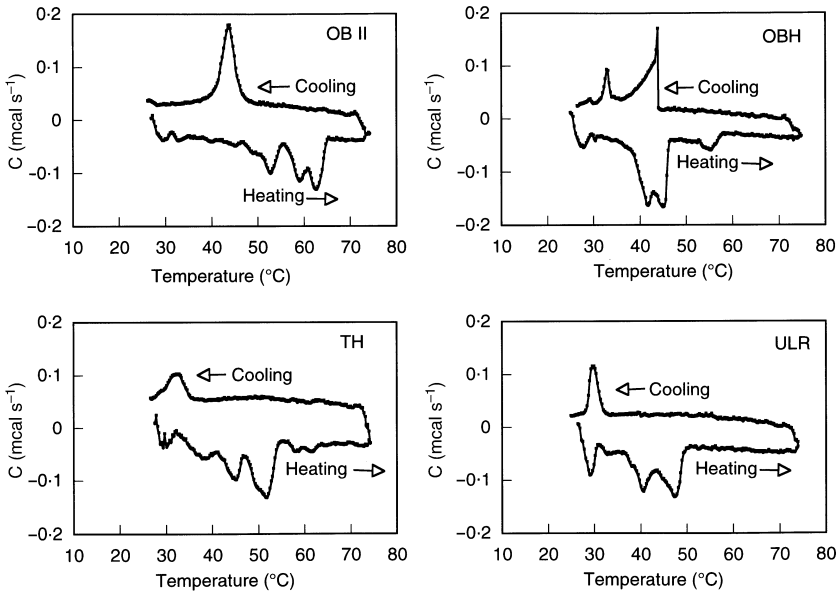

Fig. 4. Variation of Quantity of heat (c) with temperature of four materials (heating and cooling rate:0.5°C/min).

points, (b) a melting transition was clearly observed, and the setting point was somewhat lower than the melting point for three of the materials (OB II, TH, ULR) (Fig. 2 and Table 2). The mechanical properties of amorphous polymers are mostly dependent on the average molecular weight and molecular weight distribution. In the amorphous phase at temperatures higher than the melting point, the Gr(0) values of the four materials were largely different. This indicates differences in average molecular weight and molecular weight distribution of the polymer for each material as well as that in the amounts and nature of the resin or wax contained in each material, and it is supposed that the average molecular weight of OB II may be the highest and that of ULR the lowest. Another characteristic of (b) is supposed to belong to crystalline thermoplastic polymers (13, 14). Previously, employing X-ray methods, the degree of crystallinity of Tjipetir gutta was found to be 55–60% (15), and the crystal structure of pure gutta percha was reported in detail (16, 17). Moreover, regarding gutta percha endodontic points, the mechanical properties were indicative of a partially crystalline viscoelastic polymeric material in tension (9). The shear modulus of gutta percha for root canal filling obtained in our study also indicated the behaviour of a crystalline polymer.

The behaviours of OBH's Gr(0) and specific volume change were different from other materials; the variations of Gr(0) and specific volume change at heating and cooling were almost identical (Figs 2 and 3), and peaks were obtained at the same temperature on heating and cooling at DSC (Fig. 4). Also, the specific volume of OBH was the lowest. Perhaps OBH contains a large amount of plasticizer and filler rather than other materials in order to weaken the intermolecular force and to achieve a low melting point.

The advantage of crystalline polymer is that it is more stable than an amorphous polymer, but a larger amount of shrinkage occurs at phase transition. This case was applied to gutta percha for root canal filling. During the setting process, the crystallization of gutta percha is thus thought to adversely affect the sealing ability in the root canal space. The main cause of endodontic treatment failure is generally said to be incomplete sealing of the root canal, and accordingly it is important to obturate the root canal closely. Fluidity and melting point can be altered by arranging the content, the average molecular weight and molecular weight distribution, but a large amount of volumetric shrinkage with crystal growth cannot be avoided when using gutta percha.

The technique using melted gutta percha alone may not be favourable compared with conventional lateral condensation because melted gutta percha undergoes a large amount of shrinkage during setting.

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