Mechanism of Anisotropic Dimensional Changes in Wax Patterns Prepared by the Softened Wax Technique

II Determination of Paraffin Crystal Size

Kouichi WATANABE, Seigo OKAWA, Osamu MIYAKAWA, Syuji NAKANO
and Nobuhiro SHIOKAWA

Department of Dental Materials and Technology, School of Dentistry, Niigata University Gakko-cho-dori
2, Niigata City, 951, Japan

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To estimate the paraffin crystal size, small-angle x-ray scattering has been conducted. For the specimen compressed uniaxially, a very long period (155Å) has been found which is interpreted as a lamellar thickness. Moreover, this thickness remains unchanged during the recovery process.

Key Words: Small-angle x-ray scattering, paraffin crystal size, recovery mechanism

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INTRODUCTION

In the first paper of this series,¹⁾ one of the present authors has reported many experimental results about wax distortion caused by the recovery phenomenon and has discussed its mechanism. Briefly, if wax (paraffin) is deformed plastically in the softened state, transformation appears to be a smooth transition from an isotropic distribution of crystal orientation to an arranged structure. For example, under uniaxial compression, the *c*-axes of the paraffin crystals become rearranged parallel to the direction of compression and their *a*- and *b*-axes become randomly oriented in the plane normal to that direction. During the recovery process, the preferentially oriented specimen expands greatly in the direction parallel to the molecular chain axis (i.e., the *c*-axis) and shrinks to a great extent in the direction normal to that. The recovery phenomenon is caused by neither crystal reorientation nor lattice shift of the crystal. Assuming a disc-like crystal (lamella), he has proposed a crystal rotation model for the plastic deformation and a crystal slide model for the recovery phenomenon.

The purposes of the work reported here have been to justify his assumption for the crystal morphology and to present evidence for his models which account for the preferred orientation mechanism and recovery phenomenon. Therefore, it is desirable to have knowledge of the supercrystal structure. Since small-angle x-ray scattering (SAXS) is currently the only method that can be used for this purpose, this technique has been used.

In the course of conducting the experiments, it has become clear that the paraffin crystal is a lamella having a thickness of 155 Å and is stacked by uniaxial compression; also, the lamellar thickness remains unchanged during the recovery process. In conclusion, the models proposed in the previous paper are successfully confirmed in this communication.

MATERIALS AND METHODS

1. Sample preparation

Using commercial paraffin (melting range: 56~58°C), two cylindrical samples, 25 mm in diameter and 25 mm in height, were prepared by the molten wax technique and annealed at 50°C for 1 hour. A plate specimen, 3 mm in thickness, was cut out from one sample (the annealed specimen). The other was heated by direct immersion in warm water (45°C) and instantly pressed uniaxially into an 8 mm thick circular plate at a crosshead speed of 500 mm/min using a testing machine.* This specimen was subsequently quenched in ice water, wiped dry, and stored at room temperature. A plate specimen, 3 mm in thickness, was cut off from the part near its center (the compressed specimen). The recovered specimen was prepared from the compressed specimen by means of direct immersion in warm water (50°C) for 2 hours.

2. Wide-angle x-ray scattering (WAXS)

Wide-angle x-ray scattering photographs were taken by means of a point-collimation camera,[†] equipped with a flat-plate cassette. The specimen-to-film distance was 60 mm. The Cu x-ray tube was operated at 30 Kv and 20 mA. The exposure time was 3 hours. The Ni filter was not used.

3. Small-angle x-ray scattering (SAXS)

In order to investigate the supercrystal structure of paraffin, we carried out small-angle x-ray measurements with a Kratky camera set for the photographic technique. The U-type

Figure 1 Kratky U-slit system. (a) General view (b) Schematic illustration of x-ray beam trajectory

* Autograph IS 5000, Shimadzu Seisakusho Ltd., Kyoto, Japan

† Laue camera Cat. No. 1121, Rigaku-Denki Ltd., Tokyo, Japan

slit shown in Figure 1 is of our own making. A diaphragm, D_2 , lies across the gap between the two ends of the U-shaped body. The incident x-rays passing through the slit, S , cross the space between the edge, E , of the surface of the U body and diaphragm, D_2 , and strike the specimen, where they produce small-angle scattering. If the surface of the U body, the bottom surface of D_2 , and the top surface of the beam stop are located in the same plane, the background scattering from the U slit is practically negligible. The useful small-angle scattering is recorded only on one side of the slit, but a very fine linear x-ray beam is available.

Nickel-filtered $\text{CuK}\alpha$ radiation was used. The working conditions were 40 Kv and 15 mA. The incident beam breadth was 0.1 mm at the specimen, and the specimen-to-film distance was 350 mm. The exposure time was about 150 hours. Measurements were consequently possible at a scattering angle as small as 0.2° (2θ), which corresponds to an interplanar spacing of 440 Å.

4. Line broadening

In order to estimate the width of the lamella, wide-angle x-ray line broadening data from an annealed paraffin specimen were obtained by diffractometer techniques. Experimental parameters are given in Table 1. To avoid the influence of $\text{K}\alpha_1$ - $\text{K}\alpha_2$ doublets, $\text{CuK}\beta$ radiation was used. The instrumental standard was derived from measurements on annealed cristobalite powder ($>0.5\mu$) under identical experimental conditions.

The half-value breadths of the diffraction lines were used, and the following Scherrer formula²⁾ was applied;

$$L_{hkl} = \frac{0.9\lambda}{B \cos \theta} \quad (1)$$

where λ is the wavelength of the x-rays, θ is the Bragg angle of (hkl), and L_{hkl} is the dimension of the crystal normal to the (hkl) plane. B is the half-value breadth obtained from the following equation, where we assume that the x-ray diffraction profiles are Gaussian:³⁾

$$B^2 = B_p^2 - B_c^2 \quad (2)$$

where B_p and B_c are the half-value breadths of the paraffin and instrumental standard, respectively.

5. Dimensional changes caused by recovery

A cylindrical sample of paraffin, 25 mm in diameter and 25 mm in height, was prepared by the molten wax technique and annealed at 50°C for 1 hour. The sample was uniaxially compressed in the same manner as the preparation of the x-ray diffraction specimen. The

Table 1 Operating conditions of x-ray measurements for obtaining peak profiles

Target	Cu
Voltage, Current	40Kv 15mA
Full scale	2000 cps
Slit	$1/6^\circ$, 0.15 mm, $1/6^\circ$
Scanning speed	1/16 /min
Time constant	4

specimen, 5 mm $\times$ 5 mm and 3 mm in thickness, was cut off from the part near the center of the compressed sample. As a recovery treatment, the specimen was annealed by direct immersion in warm water at 50°C for 2 hours. Before and after such treatment, each dimension was measured at room temperature by means of a micrometer.

RESULTS

1. Whole SAXS pattern from the annealed specimen

The whole SAXS pattern from the annealed specimen is shown in Figure 2, which indicates that the crystals are randomly oriented. The (001) and (002) diffraction lines may be clearly noted on both sides of the beam stop. The original beam is drawn after consideration of the positions of the (00 l) lines. The diffuse scatterings on the upper side (arrow u) and lower side (arrow d) of the beam stop originate from the specimen and U slit, respectively. The line width of the (001) in the horizontal direction (L_w) is caused by the original beam shape. Accurate information, therefore, is limited to the meridional direction through the center. For this reason, we show later an enlarged portion of each diffraction pattern made by SAXS. A detailed discussion about this scattering pattern will be given later.

2. WAXS pattern from the compressed specimen

A WAXS pattern from a uniaxially compressed specimen is shown in Figure 3. The specimen has been compressed in the equatorial direction in this figure. In the previous

Figure 2 Whole SAXS pattern from an annealed specimen. u: diffuse scattering on upper side of beam stop, originating from specimen. d: diffuse scattering on lower side of beam stop, originating from U-slit. L_w : line width.

Figure 3 WAXS pattern from a compressed specimen; the direction of compression is horizontal in this figure

paper,¹⁾ we discussed in detail the relationship between the direction of compression and the appearance of $(hk0)$ and $(00l)$ diffraction maxima. That is to say, when the specimen is compressed uniaxially, the c -axes are rearranged parallel to the direction of compression, and the a - and b -axes are randomly oriented in the plane normal to the c -axes. The WAXS pattern indicates the crystal orientation clearly but gives little information about the super-crystal structure (crystal size and texture).

3. SAXS patterns under various conditions

In Figure 4, SAXS patterns which have been enlarged partly are shown. The upper one (A) is the pattern obtained from the annealed specimen and is identical with that in Figure 2. The middle two, (B) and (C), are patterns from the compressed specimen and correspond to directions indicated by arrows 'H' and 'V' in Figure 3, respectively. The bottom pattern (D) is shown as an instrumental standard in which case there is no specimen.

In pattern A, the (001) and (002) diffractions are seen clearly, which indicate the mean molecular length of the paraffin used in this study. The interplanar spacing calculated from them is 39 Å, which is in good agreement with the value obtained by means of diffractometer techniques.¹⁾ The diffuse diffraction adjacent to the beam stop originates from the specimen itself because an extremely faint diffraction can be hardly noticed in the corresponding region of pattern D. The reasons for such a vague diffraction are both the random crystal orientation and the original beam shape (line). Moreover, in the region of $0.5\sim 0.6^\circ$ in this diffuse diffraction, the arc-like diffraction (indicated by arrow) can be noticed faintly. This

Figure 4 Enlarged portions of SAXS patterns. A: an annealed specimen. B and C: compressed specimens. D: instrumental standard (no specimen). The arrows, H and V, correspond to the directions, H and V, in Figure 3, respectively.

is an important fact when considering the mechanism of plastic deformation.

In pattern B, the (001) and (002) diffractions are more clearly seen. The diffraction angles and resulting interplanar spacing are identical with those of the annealed specimen (pattern A). These findings are in good agreement with the results obtained by means of the WAXS in the previous report.¹⁾

The maximum at 0.57° can be also observed clearly in the very small angle region. This is found to be a first order diffraction because there is no maximum in the smaller angle region. This diffraction can be interpreted as corresponding to the Bragg reflection for the average distance between layers because the SAXS is for the most part dependent upon the geometrical structure of the minute, nonhomogeneous zones. In the case of paraffin, the

crystal regions are separated by amorphous (less dense) ones. From this fact, we reasonably conclude that the observed long-spacing ($155 \text{ \AA}$) corresponds to the thickness of the disk-like crystal (lamella). As a result, the lamellar thickness is four times as long as the mean molecular length. This conformity implies that molecular chain tilting does not occur in the lamella, and the amorphous layer is very thin in the direction of compression. Consequently, the compressed specimen is thought to have a periodic structure of alternating regions, crystalline (lamellar) and amorphous, as illustrated in Figure 5.

In small angles ranging from 0.57° to 1.2° , wide-spread diffraction can be seen. This diffraction can be interpreted as a second order one. According to x-ray diffraction theory, when the degree of distortion of the period is high, only the first or at most second order diffraction will be even vaguely discernible. Therefore, this SAXS pattern suggests that the values of lamellar thickness are distributed in a certain range.

In pattern C of Figure 4, we see no diffractions except the very faint (001). This fact implies that the long period in the range from $40 \text{ \AA}$ to $440 \text{ \AA}$ does not exist. In other words, the width of the lamella is larger than $440 \text{ \AA}$. But an accurate value can not be determined from this measurement. In conclusion, it is confirmed that the thickness of the crystal is much smaller than its lateral dimension.

4. Line broadening of peak profiles

In Figure 6, profiles of paraffin $(110)K_\beta$ and cristobalite $(101)K_\beta$ diffractions are shown. The half-value widths of these profiles are $5.6 \times 10^{-3} \text{ rad}$ (paraffin) and $3.8 \times 10^{-3} \text{ rad}$ (cristobalite). The resulting value depending on the paraffin crystal itself is $4.0 \times 10^{-3} \text{ rad}$. From calculations using equation (1) (Materials and Methods), the width of the crystal normal to the (110) plane is estimated as $320 \text{ \AA}$. This value contradicts the results obtained from the SAXS analysis. We intend to discuss this problem later.

5. Dimensional changes caused by recovery

As for dimensional changes caused by recovery, a large expansion (18%) occurred in the direction of the compressed axis and large shrinkage (7.2%) occurred in the direction normal to that. In other words, the uniaxially compressed specimen expands greatly in the direction parallel to the molecular chain axis and shrinks to a great extent in the direction

Figure 5 Schematic illustration of the preferred orientation of the compressed specimen

perpendicular to that.

6. Lamellar thickness of the recovered specimen

In Figure 7, a SAXS pattern from the recovered specimen (E) is shown, which was taken with an exposure time of about half that of the others. In order to estimate easily the

Figure 6 Line profiles of annealed paraffin and the cristobalite

Figure 7 Enlarged portions of SAXS patterns. B: compressed specimen identical with pattern B in Figure 4. E: specimen which has been compressed uniaxially and then subjected to recovery treatment.

effect of recovery, we show the pattern of the deformed specimen (B) again, that is, the specimen before recovery treatment. In the small angle region ($2\theta < 1^\circ$), the position of the maximum in question scarcely changes. This fact indicates directly that the average distance between layers remains almost unchanged during the recovery process.

DISCUSSION

1. Results obtained in previous studies

The paraffin used in this study is a mixture of normal hydrocarbons containing 20 to 35 carbon atoms. A small quantity of branched hydrocarbons has also been observed.¹⁾ The structure of commercial paraffin is commonly described by reference to its crystalline and amorphous components. Its crystallinity is 87% at 10°C and gradually decreases with increasing temperature in the case of paraffin (melting range 60~62°C).⁴⁾

The paraffin molecule takes a random coil conformation in the molten state even in the case of low molecular weight compounds, such as $n\text{-C}_{16}\text{H}_{34}$ or $n\text{-C}_{36}\text{H}_{74}$ ⁵⁾; but in crystalline form, it remains an extended chain in an all-trans conformation. The paraffin crystal at room temperature shows an orthorhombic symmetry, and at the solid-solid phase transition point (about 35°C), the crystal structure is transformed to a hexagonal symmetry.⁶⁾ This transition is caused by paraffin molecules tending to rotate about their chain axes and having the symmetry of a circular cylinder. The long spacing (001) of the crystal has been estimated as 39 Å from x-ray diffraction studies at room temperature and remains unchanged above the solid-solid phase transition point.¹⁾

Dawson⁷⁾ has examined the structure of a single crystal of pure n-hydrocarbon which crystallizes from a dilute solution by evaporation and has obtained some important results. His electron micrographs give evidence of spiral growth steps on the surface of a lozenge-shaped crystal, and the step distance corresponds to the molecular length.

If paraffin is deformed plastically in the softened state, the isotropic distribution of crystal orientation transforms to an arranged structure.¹⁾ In the case of uniaxial compression, the *c*-axes are rearranged parallel to the direction of compression and both *a*- and *b*-axes are randomly oriented in the plane normal to the *c*-axes. The degree of preferred orientation is associated with the crystallinity at the deformation temperature as well as the degree of deformation. On the basis of these facts, one of the authors has assumed that paraffin crystallizes as a lamella, and he has introduced the crystal rotation model to account for the mechanism of preferred orientation.

The dimensional changes due to recovery are extraordinarily large in comparison with normal thermal expansion.¹⁾ Moreover, recovery affects dimensional change as an expansion factor in the direction of the molecular axis and as a contraction factor in the direction perpendicular to that. The preferred orientation of the crystal remains unchanged during the recovery treatment. Change in its lattice constants is also negligibly small during recovery. In conclusion, the recovery phenomenon is caused by neither crystal reorientation nor the lattice shift of each crystal. Therefore, the crystal slide model has been proposed to explain the recovery phenomena.

2. Orientation mechanism caused by plastic deformation

Plastic deformation in the softened state should be discussed based on a two-phase

system composed of crystalline (rigid particles) and amorphous regions (highly viscous fluid). The interaction between the two is taken into account in terms of a friction factor. Therefore, the morphology of the paraffin crystal has an essential influence on its motion.

In this study, we have shown that the paraffin crystal has a disc-like (lamellar) shape and its thickness is 155 Å. From these results, when the crystal rotation model is adapted to the behavior of plastic deformation, one can easily understand the following mechanism. The supercrystalline structure of the paraffin is transformed from the original randomly oriented lamellae into the well-oriented stacked ones. That is, the lamellae rotate from a parallel to a perpendicular orientation to the direction of compression. A stack of such lamellae yields a very long period, and as a consequence of this, the SAXS yields a definite diffraction in the very small angle region. As has been seen in Figure 4, this is well confirmed by our experimental results.

3. Molecular alignment in the lamella

From the SAXS pattern, we conclude that the lamella thickness is 155 Å. This value is four times as long as the mean molecular length (39 Å). That is, the repeating number 'N' of the period of the *c*-axis direction is only four. On the other hand, (00 l) diffractions have been clearly detected up to the fourth order even in the randomly oriented state.⁶⁾ To discuss the relationship between repeating number and diffraction behavior, we must call attention to the Laue diffraction function.⁸⁾ According to this function, in cases where the repeating number is small, each diffraction peak becomes broadened, and high order ones can be scarcely observed. From these facts, we conclude that the lamella is composed of paraffin molecules being apparently of restricted length, and that the period of the layers is exactly constant. In the case of perfectly regular periodicity, a large number of diffractions can be detected even if the repeating number is comparatively small. For paraffin molecules to be stacked in such a regular fashion, the most natural and simplest mechanism of crystallization is a spiral (dislocation) growth mechanism not only from a dilute solution but also from the molten state. In such a case only, we can reasonably understand the precise periodicity of the stacked molecular layers.

4. Recovery mechanism

As mentioned above, the recovery becomes almost independent of both the change in lattice parameter and crystal reorientation during recovery treatment.¹⁾ Moreover, it is clear from this study that the change in lamellar thickness is too small to explain the large dimensional change. From these facts, the crystal (lamella) slide model is confirmed. While these phenomenological aspects of recovery are well-established, the essential physical character of the process (driving force of recovery) remains unknown.

We suppose that the large dimensional change by recovery is caused mainly by rubber-elasticity of amorphous molecules. This idea is an analogy taken from polymer science. However, the essential problem whether or not rubber-elasticity occurs in the case of such short molecules as paraffin confronts us. To solve this problem, we need separate information about the molecular conformation in the amorphous area both before and after recovery treatment. This approach, however, is considered very difficult because of the high crystallinity. Another approach is to clarify the relationship quantitatively between the recovery ratio and the degree of crystallinity. This is also very difficult because we can scarcely control the crystallinity of the paraffin. Though indirect evidence, if small amounts

of an amorphous substance, for example dammar, are added to paraffin, we reasonably suppose from the proposed mechanism that the extent of its recovery is larger than that of paraffin only.

5. Lateral dimension of the lamella

The lamellar thickness is successfully estimated as 155 Å. As for its lateral dimension, however, there is a serious discrepancy between the results from the SAXS analysis and that obtained from line-broadening measurements. The SAXS pattern implies that the lateral dimension of the lamella is much larger than 440 Å. In comparison to this, the line-broadening data indicate that the width of the crystal normal to the (110) plane is 320 Å. To explain this difference, we propose two ideas as follows: (1) The SAXS patterns are mainly a result of density variation. Even if the lamella is an aggregate (mosaic structure) of smaller crystallites which are separated by the crystal boundary, the SAXS pattern gives information not about the individual crystallites but about the assembly of them as one particle. Apparently, the important unit for considering the orientation and recovery mechanisms is such an aggregate because in the deformation and recovery processes it behaves as one particle; (2) Line-broadening depends upon not only the crystal size but also lattice distortion. Therefore, if appreciable distortion exists in the *a*- and *b*-axes, we would estimate the crystal size to be smaller than its true value. To distinguish the factor of the crystal size from that of lattice disorder, we must obtain information about the high order diffractions of (110). But the diffraction patterns of paraffin are too weak to permit discrimination between these possibilities. In spite of such experimental difficulties, the models to explain the orientation behavior and recovery process need not be modified at all.

CONCLUSION

Using the Kratky U-slit system and the peak profile of the WAXS, we were able to evaluate the crystal size of paraffin. The lamellar thickness is 155 Å, and its width is more than 320 Å. Furthermore, the change in lamellar thickness during the recovery treatment was negligibly small. From these facts, the preferred orientation mechanism (the crystal rotation model) and the recovery behavior (the crystal slide model) has been confirmed.

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軟化圧入法で作製したワックスパターンの異方変形の機構

第2報 パラフィン結晶サイズの測定

渡辺孝一, 大川成剛, 宮川 修, 中野周二, 塩川延洋

新潟大学歯学部歯科理工学教室

ワックスの塑性変形機構と変形回復機構を明らかにするため、クラッキーのUスリットを用いてX線小角散乱を行った。その結果、パラフィン結晶は厚さ約 155 Å の板状であることが分った。さらに、変形前はこの結晶がランダム配向であるが、一軸圧縮後、圧縮方向に明瞭な長周期 (155 Å) が現われることから、塑性変形による結晶の選択配向が枚状結晶粒の回転によるものであること

が明らかとなった。また変形回復によって、マクロ的には板状結晶の厚さ方向に18%も膨張していたにもかかわらず、板状結晶の厚さ自体にはほとんど変化が検出されなかった。この結果から、変形回復は板状結晶のすべりによるためであることがさらに確証づけられ、その駆動力としてはパラフィン非晶質のゴム弾性であると考えられる。

エラストマー印象材の硬化特性を決定するための一方法

有川裕之*, 藤井孝一*, 蟹江隆人* 上新和彦*, 井上勝一郎*, 鬼塚 雅** 自見 忠**

*鹿児島大学歯学部歯科理工学講座

**鹿児島大学歯学部歯科補綴学第1講座

印象材の操作時間、硬化時間を決定するために種々の方法が試みられてきた。そのひとつとしてウィルソン (1964) は硬化時における印象材の粘弾性変化を測定することによって操作時間、硬化時間を決定するための装置レシプロケーティングレオメータを開発した。しかし印象材の操作時間、硬化時間は粘弾性の変化よりもむしろ硬化時の弾性変化から決定するほうが臨床的にみて好ましいと思われる。そこで本研究ではそのための装置としてパルセーティングレオメータを開発した。この装置

は試料にくり返し一定ひずみを加え、その弾性回復量を測定することによって操作時間、硬化時間を決定するものである。

種々の印象材について測定をおこなった結果、本装置は操作時間、硬化時間の決定ならびに硬化時における印象材のレオロジー的性質をも知ることができるので印象材の比較検討をおこなう上で有効であることがわかった。

せっこう水和硬化体の熱的挙動に及ぼすホウ酸添加の影響

森 淑子

シドニー大学 (オーストラリア) 歯学部・補綴学教室

凝結調節剤を含有しない、二つのタイプの硬質せっこう (ハイドロカルおよびデンサイト) に2%のホウ酸を添加して作製した水和硬化体の熱的挙動を、熱膨張測定法および示差熱分析法によって検討した。

ホウ酸を添加すると、せっこう水和硬化体の 200℃

400℃間の熱収縮が抑えられ、さらに III型CaSO₄→II型CaSO₄の転移が遅延されることが明らかになった。

ホウ酸は150℃以上に加熱されると粘稠な液体となるが、このことが熱収縮の抑制や無水硫酸カルシウムの転移の遅延に寄与するものと考えられる。