Heat Curing Behavior of Light-cured Composite Resins Investigated by Dynamic Differential Scanning Calorimetry

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The heat curing behavior of light-cured restorative composite resins, light-cured crown and bridge veneering resins, pure dimethacrylate monomers, Bis-GMA monomers containing various initiators, and monomer mixtures were investigated by slow heating at a constant rate with differential scanning calorimetry (DSC) without any light irradiation—the so-called dynamic DSC measurement. Some of the light-cured restorative composite resins and the light-cured crown and bridge veneering resins showed a sharp exothermic peak due to heat curing. Pure TEDMA also showed a sharp exothermic peak, whereas the Bis-GMA monomers containing catalysts for light curing showed no exothermic peak. It seems that heat curing behavior of light-cured composite resins depends not on the decomposition of camphorquinone, but on that of the monomer itself.

Key words : Light-cured composite resins, DSC, Heat curing

INTRODUCTION

Visible light-cured composite resins have been extensively applied in modern cosmetic dentistry instead of chemically cured composite resins. Furthermore, light-cured composite resins tend to be used for the reinforcement of the mechanical properties of composite resin inlays by heat treatment. It has been previously reported that the properties of light-cured composite resins can be improved by a secondary application of heat^{1–4)}. It is thus necessary to understand the thermal polymerization behavior of light-cured composite resins to clarify why such improvements occur upon heating.

Most of visible light-cured resins contain a photosensitizer like camphorquinone, and a reducing like tertiary amine^{5–8)} which cause the initiation of polymerization by visible light irradiation. It is important to study the initial polymerization of these resins, because various properties of the set resins are doubtlessly affected by the degree of polymerization⁹⁾. The polymerization reaction of dental composite resins has been investigated by various methods including infrared spectroscopy (IR),^{10,11)} differential thermal analysis (DTA),^{12,13)} and differential scanning calorimetry (DSC)^{14,15)}. In particular, DSC can be easily used to measure the heat of polymerization, reaction kinetics, degree of conversion, and other factors for a variety of chemically cured composite resins. Moreover, the properties of dimethacrylate monomers can be measured by the isothermal^{16–19)} or heating method²⁰⁾, and the heat of polymerization of light-cured composite resins by light irradiation as well can be measured by isothermal DSC measurement^{21–23)}. We have already reported that light-cured crown and bridge veneering resins showed an exothermic peak in the DSC curve when slowly heated

from room temperature to 200°C without any light irradiation²⁴).

The purpose of this investigation is to study the heat curing of light-cured composite resins and dimethacrylate monomers by slow heating at a constant rate. Here, we employ DSC without light irradiation—the so-called dynamic DSC measurement—and discuss the heat curing behavior of commercial light-cured composite resins.

MATERIALS AND METHOD

The investigated materials, code, batch numbers, and manufacturers are listed in Table 1. Four visible light-cured restorative composite resin products, PCA, LF, OC, and PF were used. CF-C and CF-U were chemically cured composite resin pastes, catalyst and universal,

Table 1 Materials used

Type*	Materials	Code	Batch No.	Manufacturer
<i>Restorative Composite Resins</i>				
L	Photo Clearfil A	PCA	1172	Kuraray
L	Lite-fil A	LF	018559	Shofu
L	Occlusin	OC	HH13	ICI
L	Pyrorafil Light-A	PF	502	Sankin
C	Clearfil F-II Catalyst	CF-C	20043	Kuraray
C	Clearfil F-II Universal	CF-U	20043	Kuraray
<i>Crown and Bridge Veneering Resins</i>				
L	Thermoresin LC	LC	020371	GC
L	Dentacolor	DC	701023	Kulzer
L	Metacolor	MC	70401	Sunmedical
H	SR-Isosit-N	IS	6031	Ivoclar
H	Biolon	BO	20183, 080185	Caulk
H	Thermoresin	TR	140561, 220761	GC
H	Plastique	PL	058258, 078687	Shofu
<i>Monomers</i>				
	Methyl-methacrylate	MMA	C0366	Yoneyama Yakuhin
	Ethyl-methacrylate	EMA	A001	Tokyo Kasei
	Ethylene-glycol-dimethacrylate	EDMA	A003	Tokyo Kasei
	Triethylene-glycol-dimethacrylate	TEDMA	AW01	Tokyo Kasei
	Di(methacryloxy-ethyl)-trimethyl-hexamethylene-diurethane	UDMA	620908	Shin-Nakamura
	2, 2-bis[4(2-hydroxy-3 methacryloxy-propyloxy)-phenyl] propane	Bis-GMA	0416W	Shin-Nakamura
	2, 2-bis[4-methacryloxy-polyethoxy-phenyl] propane ($n=2.6$)	Bis-MPEPP	0624A	Shin-Nakamura
<i>Reagents</i>				
	Benzoylperoxide	BPO	TSR3473	Wako Pure Chemical
	Comphorquinone	CQ	FCW01	Tokyo Kasei
	Dimethyl paratoluidine	DMPT	A001	Tokyo Kasei
	Dimethylaminoethyl methacrylate	DMAEMA	A002	Tokyo Kasei
	Triethylamine	TEA	A002	Tokyo Kasei

* L : Light-cured

C : Chemically cured

H : Heat-cured

respectively. Seven crown and bridge veneering resin products were used ; LC, DC, and MC were visible light-cured resins, and IS, BO, TR, and PL were heat-cured ones. The composition of these commercial products is given in Table 2 from references²⁵⁻²⁹.

Table 2 Composition of materials (wt%)

Materials	Monomer	Reference No
<i>Restorative Composite Resins</i>		
PCA	Bis-GMA (67), TEDMA (21)	25, 26, 27
LF	UDMA, TEDMA	25, 26
OC	UDMA, TEDMA	25, 26
PF	Bis-GMA	26
CF-C	Bis-GMA (18.5), TEDMA (6.3), BPO	28
CF-U	Bis-GMA	28
<i>Crown and Bridge Veneering Resins</i>		
LC	UDMA (12), NPGDMA (9)	29
DC	UDMA (50), TEDMA (7)	29
MC	UDMA (78)	29
IS	UDMA (46), 1, 4 BuDMA (11)	29
BO	MMA (70), TEDMA (17), EDMA (11)	29
TR	Bis-MPEPP (100)	29
PL	TEDMA (70), Bis-MPEPP (30)	29

Four types of monomers were used without any additives and purification. Furthermore, 8 kinds of mixtures of Bis-GMA and catalysts were prepared : A, 2 wt% BPO ; B, 2 wt% CQ ; C, 2 wt% DMPT ; D, 2 wt% DMAEMA ; E, 2 wt% TEA ; F, 2 wt% CQ+2 wt% DMPT ; G, 2 wt% CQ+2wt% DMAEMA ; H, 2 wt% CQ+2 wt% TEA. Seven kinds of monomer mixtures were prepared from "as received" monomers : I, UDMA-10 wt% TEDMA ; 058L, Bis-GMA-30 wt% TEDMA ; M, Bis-GMA-10 wt% MMA ; N, Bis-GMA-10 wt% EMA ; O, Bis-GMA-10 wt% EDMA.

A differential scanning calorimeter* was used to make measurements. Small samples (30-40mg) of each material were weighed out into an aluminum sample pan. The samples were then slowly heated at a constant rate (10°C/min) from room temperature to 200°C. DSC range was ± 16 mcal/s. The experiments were conducted under ordinary atmospheric conditions and the sample pan was not hermetically sealed.

RESULTS AND DISCUSSION

Figure 1 shows the change in DSC curves during the heating of restorative composite resins. In the case of visible light-cured composite resins, PCA showed a sharp exothermic peak at around 110°C, and PF showed a broad exothermic peak around 160°C, while LF and OC showed no exothermic peak. After measurement, PCA set, but LC, OC, and PF did not. Therefore, it seems that this exothermic peak of PCA depended on the heat of polymerization. Chemically cured resin was not mixed. CF-C, the catalyst which contains BPO for the

* Rigaku, standard model, Tokyo, Japan

initiation of radical polymerization, showed a sharp exothermic peak around 100°C. It is clear that this peak depended on the heat of polymerization initiated by heat decomposition of BPO.

CF-U, the universal paste which contains tertiary amine for acceleration of decomposition of BPO, showed no exothermic peak.

Figure 2 shows the change in DSC curves during the heating of crown and bridge veneering resins. In the case of visible light-cured resins, MC showed a sharp exothermic peak around 130°C, and LC showed 2 broad peaks around 150°C and 180°C, and DC showed a broad peak. After the measurement, MC set, while LC and DC set only slightly. Thus, it seems that these exothermic peaks were due to the heat of polymerization. On the other hand, the thermally cured resins, IS, BO, TR, and PL showed a sharp exothermic peak around 100–120°C. The peak for IS was smaller than that of the other thermally cured resins, because it contains a large amount of filler and a relatively small amount of monomer, mainly UDMA, and the other thermally cured resins contain no filler, as shown in Fig. 2.

Figure 3 shows the change in DSC curves during the heating of 4 monomers without any kind of additives. A relatively large exothermic peak was observed around 150°C in the DSC

Fig. 1 Change in DSC curves during heating of restorative composite resins. Solid lines show visible light-cured resin pastes and broken lines show chemically-cured.

curve for TEDMA. Bis-MPEPP showed 2 exothermic peaks around 160°C and 180°C. UDMA had an exothermic peak around 170°C. However, Bis-GMA had no exothermic peak in this temperature range. Furthermore, after the measurement, the sub-surface of TEDMA in the sample pan had set, though the surface layer of the monomer had not due to inhibition by atmospheric oxygen. The setting behavior of Bis-MPEPP and UDMA were nearly identical to TEDMA, but these monomers had a relatively low extent of polymerization compared with that of TEDMA. It seems that peak depended on the heat of polymerization, because the sample hardened after the measurement. The peak height, area, and temperature of the exothermic peak in the DSC curve for TEDMA changed with each brand and lot.

Miyazaki *et al.*¹⁷⁾ demonstrated that both the rate and extent of polymerization of Bis-GMA were lower than TEDMA, UDMA and Bis-MPEPP in isothermal bulk polymerization. They explained that one reason for this low polymerization of Bis-GMA is inhibition by atmospheric oxygen. Furthermore, with respect to inhibition of polymerization they found monomers containing long-chain members between 2 functional groups and those of high viscosity to be relatively few¹⁸⁾. We found that increasing amounts of TEDMA in monomer mixtures of Bis-GMA and TEDMA decreased the conversion of polymerization in air, since the rate of oxygen diffusion in a resin liquid increases with decreasing viscosity¹⁶⁾. Thus,

Fig. 2 Change in DSC curves during heating of crown and bridge veneering resins. Solid lines show visible light-cured resin pastes and other lines show heat-cured.

although TEDMA is strongly inhibited by atmospheric oxygen, TEDMA showed a sharp exothermic peak. It thus seems that the heat curing behavior of dimethacrylate monomers depends on their chemical properties.

Many monomers appear to undergo spontaneous polymerization when heated in the apparent absence of catalysts. In most cases, the observed polymerizations are initiated by the thermal or photolytic homolysis of impurities (including peroxides or hydroperoxides formed due to O_2) present in the monomer. Most monomers when exhaustively purified (and in exhaustively purified reaction vessels) do not undergo a purely thermal, self-initiated polymerization in the dark³⁰. Therefore, it seems that TEDMA showed spontaneous polymerization initiated by thermal homolysis of a peroxide due to O_2 .

Figure 4 shows the change in the DSC curves of 8 kinds of mixtures of Bis-GMA and catalysts. Sample A, Bis-GMA with 2 wt% BPO, showed a large exothermic peak, apparently due to the heat of polymerization. Sample C, Bis-GMA with 2 wt% DMPT, showed a broad exothermic peak. Other mixtures showed no exothermic or endothermic peaks. It was found that a photoinitiation reagent like CQ and combination of CQ and various amines had little potential for heat curing. Therefore, although OC has a relatively high CQ content among these restorative composite resins (OC, 1.03 wt% ; PCA, 0.83 wt% ; LF, 0.35 wt%)⁷,

Fig. 3 Change in DSC curves during heating of 4 kinds of monomers without any additives.

OC showed no change in the DSC curve from room temperature to 200°C as shown in Fig. 1. Taira *et al.*⁹⁾ reported that LF and OC contain dimethyl-aminoethyl methacrylate (DMAEMA), but PCA contains an another undetected additive as a reducing agent. It is unclear whether the sharp exothermic peak in the DSC curve for PCA depends on the heat of polymerization initiated by the combination of CQ and an unknown reducing agent. Therefore, it is concluded that camphorquinone can not be accelerated by the application of heat.

Figure 5 shows changes in the DSC curves of 7 kinds of monomer mixtures without any catalysts. Pure TEDMA showed a sharp exothermic peak around 150°C, as shown in Fig. 3. Monomer mixtures I, J, K, and L containing TEDMA showed exothermic peaks corresponding to each major monomer except TEDMA. Monomer mixtures M, N, and O containing MMA, EMA, and EDMA showed no exothermic peaks in this temperature range. These monomers seem to evaporate during the measurement because of low boiling point (MMA, 100°C; EMA, 30°C; EDMA, 98°C). Therefore, it seems that thermal behavior of monomer mixtures depends on that of the monomer and there is little change due to

Fig. 4 Change in DSC curves during heating of 8 kinds of mixtures of Bis-GMA and catalysts: A, 2 wt% BPO; B, 2wt% CQ; C, 2wt% DMPT; D, 2 wt% DMAEMA; E, 2 wt% TEA; F, 2 wt% CQ+2 wt% DMPT; G, 2 wt% CQ+2 wt% DMAEMA; H, 2 wt% CQ+2 wt% TEA.

co-polymerization.

In the case of light-cured restorative composite resins, although PCA, LF and PF contain TEDMA, only PCA showed a sharp exothermic peak in the DSC curve. In the case of light-cured crown and bridge veneering resins, MC showed a sharp exothermic peak in the DSC curve. Although DC also contains TEDMA, it has no exothermic peak. Thus, it seems that the exothermic peaks of the DSC curve for light-cured commercial resins during heating depend on the heat of polymerization of each monomer itself contained in the resins, or the decomposition of heat-accelerated catalytic impurities in the manufacturers' products. Dynamic DSC measurement seems to be an adequate method for evaluation of the possibilities of heat curing of light-cured composite resins, and for discussion of the monomer composition and impurities of the monomers.

CONCLUSION

The heat curing behavior of light-cured restorative composite resins, light-cured crown

Fig. 5 Change in DSC curves during heating of 7 kinds of monomer mixtures without any catalyst: I, UDMA-10 wt% TEDMA; J, Bis-MPEPP-10 wt% TEDMA; K, Bis-GMA-10 wt% TEDMA; L, Bis-GMA-30 wt% TEDMA; M, Bis-GMA-10 wt% MMA; N, Bis-GMA-10 wt% EMA; O, Bis-GMA-10 wt% EDMA.

and bridge veneering resins, pure dimethacrylate monomers, Bis-GMA monomers containing various catalysts, and monomer mixtures were investigated by slow heating at a constant rate with differential scanning calorimetry (DSC) without any light irradiation—the so-called dynamic DSC measurement. Some of the light-cured restorative composite resins and the light-cured crown and bridge veneering resins showed a sharp exothermic peak around 110–130°C due to heat curing. Pure TEDMA also showed a sharp exothermic peak around 150°C, whereas the Bis-GMA monomers containing catalysts for light curing showed no exothermic peak. It seems that heat curing behavior of light-cured composite resins depends not on the decomposition of camphorquinone, but on that of the monomer itself. It also seems that dynamic DSC measurement is an adequate method for evaluating heat curing of light-cured composite resins, and for discussion of the monomer composition and impurities in monomers.

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Dentin primer 塗布によるウサギの皮膚に対する病理学的変化の研究

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レジンの象牙質に対する接着性を改善するために用いられる dentin primer によって、術者の手指を主とする皮膚組織に引き起こされる副作用を検討するために、glutaraldehyde (GLU) または HEMA の水溶液、およびこれらの混合水溶液からなる試作 primer の 7 日間の反復塗布によって、ウサギの皮膚に見られる変化を病理学的に検討した。さらに、新たに試作した dimethylaminoethyl methacrylate (DMAEM) 水溶液の接着性を検討するとともに、この水溶液による副作用も合わ

せて検討した。その結果、DMAEM は従来報告されている、HEMA および GLU を含む水溶液と同等の接着性を示したものの、ウサギの皮膚に対して強力な炎症作用を引き起こし、さらに、このような炎症は、塗布 7 日後にも完全に回復することは出来なかった。さらに、GLU を含む水溶液でも同様の炎症が惹起されたが、その程度はやや軽微で、7 日後にはほぼ完全な回復が観察された。これに対して HEMA および glyceryl methacrylate の塗布では、著明な変化は全く認められなかった。

DSC 昇温法による光重合型コンポジットレジンの熱重合の研究

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成形修復用光重合型コンポジットレジン, 歯冠修復用光重合型硬質レジン, 純粋ジメタクリレートモノマー, 触媒を添加した Bis-GMA, モノマー混合物の熱重合挙動を、走査型示差熱量測定 (DSC) により、光照射せずに、一定の昇温速度でゆっくり過熱した場合、すなわち DSC 昇温法により測定した。成形修復用光重合型コンポジットレジンおよび歯冠修復用光重合型硬質レジンの中

には、大きな発熱ピークを示すものがあった。また、純粋 TEDMA モノマーも大きな発熱ピークを示した。一方、カンファーキノンを添加した Bis-GMA は発熱ピークは生じなかった。ゆえに、光重合型コンポジットレジンの熱重合挙動はカンファーキノンの熱分解によるものではなく、レジン成分であるモノマー自体の熱重合によるものと考えられた。

HEMA 水溶液の象牙質知覚鎮静効果に関する臨床的評価

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象牙質知覚過敏症に対する 35 % HEMA 水溶液の鎮静効果を臨床的に評価するために、歯周治療を要する 48 患歯の歯面を EDTA によって清掃し、35 % HEMA 水

溶液を塗布して市販の dentin bonding agent で被覆した。このような手法による知覚鎮静効果は、圧搾空気、冷水、および探針によって歯面を刺激した際の反応を、