ABSTRACT

DYNAMIC MECHANICAL ANALYSIS OF PARTICULATE DENTAL COMPOSITES

by
Minu Parekh

Typically, dental composites are used in different configurations and situations. When a cavity forms at the occlusal surface of a posterior tooth, a class I and class II filling is used, depending on the extent and nature of the cavity formed. These fillings have to be designed to resist mechanical abrasion and occlusal stress during chewing, bruxing and other tooth functions. Class IV fillings are also designed to resist biting stress. In these applications composites with high filler loading with filler particles of size >0.6μm are used. These composites are typically known as minifill or midifill composites. When more than one particle size range is used, they are also refereed to as hybrid composites. When the restorations are prepared on interior tooth surfaces not subject to direct application of occlusal or biting stresses (e.g., class III and class V fillings), the composites are designed with less emphasis on the abrasion resistance and mechanical properties of the composites.

Typically, microfill composites with limited filler loading of colloidal silica (of 0.4μm size) is used for such applications. It has been reported in recent years that these fillings must be designed to flex with tooth function and hence should possess lower modulus of elasticity. The posterior restorations, on the other hand, must be sufficiently stiff to resist masticatory stresses. For these reasons, dynamic mechanical properties of minifill/midifill and microfill composites need elucidation. The dynamic mechanical response is best

studied under flexural mode of dynamic deformation. Among the properties considered important for composite resins are viscoelastic properties such as storage modulus (E′), loss modulus (E″) damping, glass transition, etc. In this study dynamic mechanical analysis using flexural mode of deformation in the temperature range of -50 to 180°C has been used to characterize the viscoelastic properties of four composites at 37°C with visible light cure.

The results indicate that hybrid composites have higher viscoelastic properties than the microfill system. The resin is characterized by higher storage modulus mode and loss modulus across the entire range of temperature investigated. The results indicate that the filler loading and cross linking effects may be responsible for the variation of viscoelastic properties as a function of different variables (e.g. filler loading, particle size).

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CHAPTER 1

INTRODUCTION

Synthetic resins have been developed as restorative materials principally because of their esthetic properties. The early resin restorations were made by cementing heat cured acrylic inlays or crowns into the prepared cavity. However the low modulus of elasticity and lack of dimensional stability of the resin invariably resulted in a fracture of cement with subsequent leakage and failure of the restoration.

The development of the self curing acrylic materials in the late 1940's made feasible the direct restoration of the teeth with resin. The monomer and polymer could be combined and the resultant dough or gel inserted into the prepared cavity, where it polymerized in situ. The use of acrylic resin for tooth restorations has been the subject of much controversy. Certain properties such as esthetic qualities and insolubility made it superior to silicate cement.

Advancement in polymer research have resulted in the evolution of improved resin systems for use as restorative materials which would also adhesively bond to tooth structure. Thus two kinds of direct filling resins are currently in use, although there is a strong preference for the composite resins. The unfilled resins are classified as type I and composite resins are type II. There are several classifications of composite resins, the so called "conventional" composite and newer mini,mide and microfilled resins. In the microfilled resins (composite) the amount of filler is much less than in the type II resins (composite) and the resin matrix of the latter is also different.

Type II direct filling resins stipulates a working time of at least 1.5 minutes and a

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maximum hardening time of 8 minutes, just as it does for type I resin. However, the requirements for other properties such as diametrical tensile strength and watersorption, are more stringent for the type II resin. It is obvious that the composite resins are superior to the unreinforced acrylic resins, in respect to most mechanical and physical properties.

This would be anticipated because of the strengthening effect of the filler and the difference in the properties of the resin matrix. The molecular weight of the BIS-GMA molecule is approximately 512, while that of the methyl metacrylate monomer is only 100. Because of the higher molecular weight of the monomer and high concentration of inorganic filler, the polymerization shrinkage of approximately 1.4% for the conventional composite is much less than that of the unfilled acrylic resin, which is in the range of 7%.

Thus the conventional composite resins should have less tendency to pull away from the walls of the cavity. For the same reason the coefficient of the thermal expansion is appreciably lower than that of an unfilled acrylic resin. The linear coefficient of the thermal expansion of the conventional resins is approximately 37×10^{-6} as compared to 92×10^{-6} for unfilled acrylic resin. Watersorption of the conventional composite is also less than that of unfilled acrylic resins.

The conventional composite resins are appreciably stronger than unfilled resins when loaded in compression. The composite also has a higher tensile strength. The fatigue limit is approximately 65% of the compressive strength. The conventional composite resins also have a much higher modulus of elasticity than the unfilled acrylic resins. This would suggest that the stiffer material would be less susceptible to elastic deformation when subjected to masticatory force. The composites are much harder than the unfilled acrylic resins. In general the properties of these "composite" resins are

superior to those of the conventional acrylic resin. There is a need to characterize the dynamic mechanical properties of composites.

1.1 Objective

There is a need to characterize viscoelastic properties such as storage modulus, loss modulus, Tan Delta, Glass transition temperature, etc, of typical denture base composite materials.

The objectives of this investigation were:

1. To characterize the dynamic mechanical properties of the currently available commercial composite systems, with special emphasis on the different filler materials of composites i.e.

2. To determine the glass transitions near and above the ambient temperatures in the oral environment and their relationships to the compositions of the formulations.

3. To understand the changes in the viscoelastic properties as a function of temperature and correlate these changes with the structures and transitions in the materials.

4. To characterize the thermal expansion variations and softening at 37°C (mouth temperature) including the glass transition in a thermomechanical analyzer (TMA) and weight loss measurements in a thermogravimetric analyzer (TGA) and correlate this information to the dynamic mechanical properties and their changes.

1.2 Matrix Material

The majority of diacrylates present in commercially available composite restorations are based on a viscous resinous component having an aromatic backbone derived from

bisphenol - A, together with one or more aliphatic mono- and/or diacrylate for the purpose of viscosity reduction and occasionally to improve reactivity with regards to a snap cure in vivo. The bisphenol A based species may be obtained from the reaction of bisphenol A or its alkylhydroxy derivatives with methyl-acryloyl chloride, from the reaction of bisphenol A with glycidyl methacrylate, or from the reaction of methacrylic acid and diglycidyl either of bisphenol A, the latter two referred to as the BIS/GMA molecule.

In the composites marketed by many companies, use is made of the BIS/GMA molecule to impart aromaticity to the resinous backbone of the composite matrix. This species contains hydroxyl groups spaced along the chain and an equal number of ether links offering some rotational freedom to the structure.

1.3 BIS-GMA

It is a resin based upon an epoxy starting material which is being used as the matrix for the commonly used composite restorative materials. The resin formulation is actually a reaction product of the methaacrylic acid and diglycidal ether of bisphenol-A. The backbone of the molecule is similar to that of an expoxy resin, but the functional reactive group in the molecule are acrylic. This resin is often referred to as BIS-GMA system.

BIS-GMA (Dimethacrylate) monomer

$$H_2C=C\ COOH_2 - C - CH_2O ---- \bigcirc - C - \bigcirc OCH2-C- CH_2OCC = CH_2$$

with substituents: O, OH above; CH_3, H below on left side; CH_3 on center; H, O above and H, CH_3 below on right side.

BIS-GMA also can be obtained by the reaction of glycidyl ether of bisphenol-A and methacrylic acid. This hybrid molecule is classified as a thermosetting methacrylic resin. It has proved to be suitable as binder for reinforcing fillers. It has a somewhat lower polymerization shrinkage than does methylmetacrylate and hardens rapidly under oral conditions.

1.4 Fillers

The designation composite for this class of dental restoratives derives from the incorporation of reinforcing fillers into the resinous matrix. In the general plastic technology, the term composite is most frequently applied to glass fiber laminates made from epoxy, polyester, acrylic and other more specialized resinous binders. In the dental technology, the reinforcing fillers are silicates. such as lithium. aluminum silicate, hydroxy apatite and various silica as well as various kinds of glass, quartz and other inorganic materials. In form they may be irregular particles, platelets, rods or breads.

Optimization is accomplished in terms of the proper fillers and blends of fillers, the proper particle size and shape and blends thereof and the proper total loading volume of the selected combination. Filler serves to modify a number of important properties. They reduce shrinkage of the composites. The reduced shrinkage leads to a distinct improvement in marginal adaptation over unfilled systems in direct proportion to the filler loading volume. Filler increases compression strength of the composite and in optimized systems they provide compressive strength at oral temperatures greater than the average for human tooth structure. Fillers generally increase hardness of composites and also influence favorably or adversely opacity, translucency and color matching.

Fillers most generally adversely influence polishability of the composites but by proper formulation the adverse influence can be minimized. Fillers reduce thermal expansion and contraction rates and while it is not possible to obtain values as low as with tooth structure, their use nonetheless results in minimum wall separation during low temperature cycling. Fillers improve stability in oral fluids to the extent that they are completely insoluble in them. Fillers may be used to provide flow control for the composites when they are employed for specialized applications. Fillers, by reducing shrinkage, permit greater retention of vander waals adhesive bonds between composites and enamel and by lowering thermal expansion rates permit better retention of the bonds with time _in vivo_. In the dental composites most commonly silicate glass filler is used.

CHAPTER 2

LITERATURE SURVEY

With the invention of the redox initiator systems for polymerization of methacrylate monomers at room temperature, it was possible to make direct filling resins. However, the initial resin-based filling material had serious defects. These materials were of insufficient technical quality to replace the fluoride containing silicate cements, despite their apparent chemical and technical defects. The subsequent development (2) and introduction on the market of composite resin restorative material, have led to a replacement of the silicate cements and unreinforced direct filling resins as filling materials in anterior teeth. The introduction of the acid etch technique has improved the quality of composite resin restorations.

During the early development, several composites were introduced for use in the posterior region. The major problems associated with these posterior composite material were secondary caries (4) and inadequate resistance to wear. Composites are not at present as resistant to wear as amalgam (5). The problem of secondary caries is, at least in part, related to the insertion technique (6).

Dental composites are complex materials, composed mainly of ceramic fillers, an organic matrix and the interface between fillers and matrix. In addition, these materials contain an initiator system, pigments and stabilizers such as inhibitors, antioxidants and UV - stabilizers. All these components are important for the properties of the polymerized composite restorations.

2.1 Properties of Composites

It has become apparent that composite restorative materials had a notable short-coming; for instance a lack of wear resistance which leads to subsequent loss of anatomic form when used for class I and II restorations. Leinfelder et al. (7) observed that in general the loss of substance was uniform, i.e. the restoration appeared to be submerged below the original surface, exposing the enamel walls of the original cavity preparation. An even more serious shortcoming as a result of poor wear resistance could be approximal "flattening" of class II composite restoration, causing subsequent loss of contact with the risk of mesical drifting of adjacent teeth (7).

Different laboratory methods of evaluating wear have been reported (8-10). All these methods are based on the assumption that wear is due to mechanical or physical influence on the material (11-13). A porous layer has been observed beneath those surfaces of composite restoration that are exposed to the oral environment. It has been suggested that this phenomenon is caused by environmental softening of the composites (14). Furthermore, it has been indicated that in vivo wear processes are partly due to hydrolytic degradation of filler particles (15,16).

2.2 The Organic Matrix

Erosion of the resin matrix and exposure of filler particles can be caused by accelerated aging using xenon light and intermittent water spray (9,17). Specimens submerged in water and exposed to xenon light with the spectral distribution of the sunlight also reveals degradation of the polymer matrix and exposure of filler particles (18). The formation of a groove between a ceramic inlay and tooth enamel caused by the disintegration of

composite resin material (19) also indicates that chemical degradation is an important factor.

In general, it can be stated that no in vitro water tests have been found that will predict composite wear performance accurately (20). This situation is due to the fact that in vivo wear is a complex process. The processes involved are probably of chemical, physico-chemical, thermomechanical and mechanical nature.

The degree of conversion after polymerization may affect the resistance to chemical degradation of resin based filling materials. Residual double bonds in polymeric materials make them less resistant to oxidative degradation reactions (21). The molecular structure of the monomers and the quantity of the different monomers, as well as the quantity and type of fillers used in dental composite materials, will affect the properties of the final, polymerized materials.

Current research focus as on the development of polymer systems with better conversion than those in the present systems. Attention is also being directed towards systems which have a structure which will prevent oxidative degradation.

The composite fillers - As described by Dr. Leinfelder and others, the types of fillers can be divided into groups according to the size of the filler particles. The tendency has been to reduce the size of the particles and to increase the filler loading in the dental composites. The first generation of the dental composites contained mainly quartz particles. Now there is a great variety in the composition of fillers. In order to delineate the composite restorations against both dentin and enamel, X-ray opaque materials have been introduced. Elements like barium, strontium, and zinc have been incorporated in the respective glasses in quantities sufficient to give radiopacity.

It is difficult to predict the optimal properties for filler particles, especially with regard to particle size. Undoubtedly, composite systems with a high filler loading($\approx$70 vol.-%) appear desirable. Fillers which are hydrolytically stable should be used in order to assure a stable filler/matrix interface.

2.3 Monomers used for the Resin Matrix

Resin-based restorative materials usually contain dimethacrylate monomers. Analyses of proprietary dental resin and composite materials have demonstrated variation in composition of such materials (22-24). Most of them contain the high-viscous and the relatively rigid aromatic monomer 2,2-bis 4-(2-hydroxy-3-methacryloylaxy-propoxy)phenyl propane (BIS-GMA). The commercially used BIS-GMA can be resolved into several components. The main components have been identified as a linear BIS-GMA and a branched BIS-GMA. The ratio of linear to branched BIS-GMA is 3:1. In some composites the presence of derivatives with a higher molecular weight than the linear and branched BIS-GMA also have been observed (25).

Other high-viscous aromatic dimethacrylate monomers similar to BIS-GMA are also used in dental composite materials (22-24,26). Such dimethacrylates are 2,2-bis 4-methacryloyloxyphenyl propane (BIS-MA), 2,2-bis 4-(2-methacryloyloxyethoxy)-phenyl propane (BIS-EMA) and 2,2-bis 4-(3-methacryloyloxypropoxy)phenyl propane(BIS-PMA).

To decrease the viscosity of the resin systems, monomers with low viscosity, such as triethyleneglycol dimethacrylate (TEGDMA) and ethyleneglycol dimethacrylate (EGDMA) are used. Other oligaethyleneglycol dimethacrylates, such as diethyleneglycol

dimethacrylate (DEGDMA) and tetraethyleneglycol dimethacrylate (Te-EGDMA) are present in trace amounts. Other types of monomers have also been identified. The filling material Visio-dispers (VD) (ESPE) contains various isomers of bis(arryloyloxymethyl)- and bis(methacryloyloxymethyl)tricyclo decane (27). Several dental restorative materials contain the diurethane dimethacrylate 1,6-bis(methacryloyloxy-2-ethoxycarbonylamino) - 2,4,4-trimethyl-hexane (UEDMA) as the only monomer, whereas other products contain this monomer in combination with other monomers, such as BIS-GMA and TEGDMA.

The very first proprietary visible light activated dental composite Fotofil (FF) (ICI) contained the monomer EGDMA together with a urethane dimethacrylate system (TUDMA) with several isomers and derivatives (23,25). This monomer system contained di(urathanophenyl)methane groups which remain as such in the polymerized material. It is known that such a system is susceptible to photo-oxidation with formation of quinoid groups (28,29). This formation of quinoid structure is accompanied by intense yellowing which can be observed with the composite FF without added UV-stabilizer (18). In the composite Occlusin (OC) (ICI) the sensitive di(urethanophenyl)methane group has been substituted by a hexamethyl-enediurethane group. The composition of OC has been determined by means of high performance gel permeation chromatography (HP-GPC) and nuclear magnetic resonance (NMR) spectroscopy (25). OC contains TEGDMA and a mixture of oligomeric urethane dimethacrylates. Other composites Nuva-Fil (NF), Prisma-Fil (PF) and Ful-fil (Fu-F) (Caulk) contain TEGDMA and a linear poly- urethane synthesized from BIS-GMA and hexamethylenediisocyanate (23,25).

When assessing clinical and biological properties, as well as laboratory data, the variations in composition and purity of commercial resin systems should be taken into

consideration. It is difficult to predict which monomer system or combination of monomers will give optimal properties for dental composite resins. Emphasis must be placed on obtaining a high degree of conversion, a highly crosslinked system and a system which is resistant to hydrolytic and oxidative degradation. "Snap set" systems are needed for dental uses, and they must have a low volume contraction during polymerization.

2.4 Initiator Systems

Most commercial, chemically activated composite materials contain the initiator benzoyl peroxide, which is activated by the tertiary aromatic amine N,N-bis(2-hydroxyethyl)-p-toluidine. The polymerization-initiating benzoyl radicals are formed by a multistep process (30).

A few commercial composite materials employed external energy in the form of ultraviolet (uv) light for activation. Upon exposure to UV light a photoinitiator, e.g. benzoin methyl ether, undergoes photofragmentation with formation of polymerization initiating radicals (31).

Within the last eight years composite resins utilizing visible light for activation of polymerization have become popular. A commonly used photoinitiator system is based on a diketone and a reducing agent, e.g. camphoroquinone and a tertiary amine like N,N-dimethylaminoethyl methacrylate (32,33). It is assumed that the diketone absorbs radiation energy and is transferred to excited states. At the appropriate excited state (triplet state) the diketone should then combine with the reducing agent to form an excited state complex (epiplex) which breaks down to give reactive free radicals (31,32,34,35).

During the initial stage of a polymerization process with methacrylate monomers,

e.g. during the working time of chemically activated restorative resins, the polymerization is inhibited by oxygen. The reactivity of oxygen to a radical is much higher than that of a monomer (36-38). Length of the inhibition period is proportional to the initial concentration of oxygen, and the added inhibitor (39,40). The concentration of dissolved molecular oxygen in liquid methacrylate monomers is about 60 ppm (37). During the inhibition period this oxygen is consumed by the radicals formed. Restorative resins in contact with air during polymerization contain unpolymerized surface layers caused by diffusion of atmospheric oxygen into the liquid resin (41,42,38). The thickness of the unpolymerized film on the surface is dependent on the viscosity of the resin and on the initiating system used. For chemically activated systems the structure of the tertiary aromatic amine is of importance. UV-light polymerized resins have thinner inhibited layers than chemically activated resins of comparable composition. This is because the rate of radical formation is much higher in UV-light activated (38). Improvement of the visible light sources is needed.

2.5 Conversion to a Polymeric System in Composites

Ten years ago the incomplete conversion in dental sealant was demonstrated (43). The degree of conversion or extent of polymerization of dimethacrylate monomers, can be determined by measuring the quantities of unreacted methacrylate groups using transmission infrared (IFk) spectroscopy and the more rapid method of Fourier Transform infrared (FTIR) spectroscopy.

After polymerization, proprietary dental sealant have different quantities of unreacted methacrylate groups (UM), i.e. they exhibit different degrees of conversion

($\%DC = 100-\%UM$). For the materials containing the aromatic dimethacrylate monomers BIS-GMA and BIS-MA as well as the low-viscous monomer TEGDMA, the quantity of unreacted methacrylate groups (UM 22 -36%, i.e. DC = 64 - 78%) could be correlated with the quantity of the rigid dimethacrylates BIS-GMA and BIS-MA. The relationship between degree of conversion and the ratio between TEGDMA and aromatic monomers of BIS-GMA based unfilled resins has been confirmed in several publications (40,44.45). The degree of conversion decreases with increasing concentration of the rigid aromatic monomers and the degree of cure increases with increasing concentration of the low-viscous TEGDMA. It has also been shown that inhibitor concentrations higher than those commonly used in resin systems have an adverse effect on conversion (40,45). More effective inhibitors also have a similar adverse effect (46). The duration of the inhibitor period (working time) is proportional to inhibitor concentration (46).

A UV-cured resin, Nuva Seal (NS) (Caulk), contains the monomer methyl methacrylate (MMA) in addition to BIS-GMA. The high conversion of 85% with this monomer system is probably due to the presence of the monomethacrylate MMA, which is small and able to diffuse and react with the active ends of the growing macroradicals even after the sealant has hardened. It has been shown that the conversion of the individual mono-, di- and trimethacrylate monomers varies (47-49). Such investigations reveal that the degree of conversion decreases with the number of methacrylate groups. The conversion is also dependent on molecular weight and molecular structure of the monomers.

It has also been demonstrated that the mechanical properties (tensile strength, compressive strength, flexural strength modules, dynamic mechanical properties and

hardness) of unfilled resins increase with increasing degree of conversion (50,51,45). With respect to degree of conversion in the organic matrix of composite materials containing the two monomers TEGDMA and BIS-GMA a similar correlation as for the unfilled resins can be made. The composite material Epolite (GC) contains TEGDMA (44 wt-%) and BIS-GMA (54 wt-%) and after the chemically induced polymerization the degree of conversion has been determined to be approximately 70%. The pastes of Concise (3M) contain TEGDMA (22-24 wt-%) and BIS-GMA (72-73 wt-%) and the degree of conversion is approximately 58% after curing. The degree of conversion is of the same magnitude for the chemically activated composites P-10 (3M) (61%) and Profile (5.5. White) (55%) recommended for posterior restorations, as for the conventional anterior composites (Ruyter and Oysaed, 1986). The poor conversion of the monomers in composite restorative materials with chemically induced polymerization has been confirmed by several research groups (52,53,24).

In restorative resins with chemically induced polymerization the reaction takes place almost uniformly throughout the bulk of the material, and curing is not generally dependent on ale thickness of the restoration. However, composites which are activated by UV or visible light polymerize only to a certain depth (54,35). The depth of cure is dependent on the depth of penetration of the activating light in the composite, i.e. the depth of cure is dependent on material composition, tight source and exposure time. Incomplete polymerization in the inner part of the restoration may lead to retention failures and also to adverse pulp tissue reactions.

By means of infrared multiple internal reflection spectroscopy (IR-MIR) the conversion in different depths of UV and visible light activated composite materials can be

determined (35). At shallow depths (less than 0.5 mm) the conversion varies for the different materials (35,25). This optimal conversion is mainly dependent on the monomer composition. The monomer systems of the visible light cured composites P-30 and P-10 with chemical initiation are almost the same. After polymerization both these posterior composites exhibit a degree of conversion of approximately 62% (18,25). It has also been reported that unfilled resins formulated with BIS-GMA and TEGDMA have the same degree of conversion irrespective of whether chemical activation or visible light activation is used (45). The light-cured posterior composite DC with the complex resin system of relatively flexible monomers and oligomers exhibits a conversion of 70% after polymerization. The oldest visible light activated material FE contains EGDMA with a short link between the two methacrylate groups and the relatively rigid TUDMA oligomer system. After polymerization this composite exhibits the low conversion of 55%. The UV-activated material Estilux Microfill (ELM) (Kuizer) was based on a relatively small amount of BIS-GMA (39 wt-%) in addition to the flexible monomers TEGDMA (33 wt-%) and UEDMA (24 wt-%) (23). The conversion in different depths shows a region with only minor changes in degree of conversion. Below a certain depth, approximately 1 mm, there is an abrupt decrease in the conversion with no polymerization below 1.3 mm when a 40 s exposure to a UV-light source is applied. The shallow curing depth of the microfill material ELM is due to light scattering of the UV-light caused by the particles in the composite. The optically effective particle size of the silica particles in ELM (approx. 0.15 μm) is close to half the wavelength of the effective activating UV-light, which is the optimal size for maximum light scattering (35). Better results with respect to curing depth are obtained for the visible light activated material Durafill (DF) (Kuizer) with practically

the same monomer system as ELM, probably because the wavelength of the effective activating visible light is more than twice the diameter of the agglomerated microfill particles. The conversion at shallow depths of DF, however, is poorer than that of ELM, 62 and 70% respectively. UV-light activation is more efficient than visible light activation. The three materials NF, PF and Fu-F all have the same resin system. The better conversion of 70% for the UV-activated material NF compared with 59% for the visible light activated anterior material PF and posterior material Fu-F is demonstrated (35,25). The reason for the better conversion with UV-light than with visible light might be that the quantum yield for radical formation by UV-light is higher than for a visible light photo initiator system.

The optimal conversion of visible light activated restorative materials also depends on the quality of the light source. The spectral distribution of several proprietary dental photopolymerization light units has been determined (55). The performance characteristics of proprietary light sources vary. The two lamps which first entered the market, the Fotofil lamp (FL) (ICI) and Translux (TL) (Kulzer), are examples of differences in light performance. The light source FL emits light in the visible part of the spectrum only, 400-550 nm. The light unit TL, however, also emits radiation below 400 nm. The degree of conversion at a depth of 0.5 mm, i.e. the region of optimal conversion varies when using these two different light sources. The optimal degree of conversion for the material Silux (3M) was 65% with the TL lamp and 54% with the FL lamp. The polymerization and therefore also the quality of the cured filling material is better with the TL lamp than with the FL lamp. Activating the composite PF revealed a 60% optimal degree of conversion with the TL lamp and 51% with the FL lamp. Similar differences were also observed for

several composite restorative materials. Such results indicate that the spectral distribution of the light is important for the degree of conversion after polymerization. It is probably crucial that there is sufficient energy in the shortwaved part of the visible light. The TL lamp emits more energy at for example 420 nm than the FL lamp.

The total curing depth after polymerization, i.e. the depth which appears hardened, varies somewhat with the different light sources. With an exposure time of 40 s the total curing depth varies between 3.6 and 4.7 mm for the material DF. Usually only 1/2 to 2/3 of the total curing depth is optimally polymerized (35), i.e. with this relatively long exposure time the optimal curing depth may vary between 1.8 to 2.4 mm for this microfill material. The depth of cure is not only dependent on the light intensity, but also on how the lamp focuses the light (56). Increasing exposure time increases the curing depth. The curing depth increases considerably with exposure times up to one minute. The pigmenting of the materials is of great importance for the curing depth. Yellow pigment absorbs shortwaved blue light, i.e. this is the light which induces the polymerization reactions. Filling materials with yellow pigments must be irradiated for a much longer time than light pigmented materials.

Visible light activated materials are sensitive to ambient light (57). Exposed to direct sunlight, they harden within 10 to 15 s (58). Because modern operating lights use quartz halogen bulbs of similar quality to the activator lamp units, the handling period of the light activated restorative materials is limited.

2.6 Mechanical Properties under Dry and Wet Conditions
Due to the complex stress situation composite restorative materials are subjected to in

vivo, it is difficult to predict which mechanical properties are the most important. In addition to the complex stress situation, the effects of the environment are important for the properties of the materials (59,60,14,61). Water sorption may affect composite materials by reducing the wear resistance (62). The water absorbed by the polymer matrix could cause filler-matrix debonding or even hydrolytic degradation of the fillers (16).

In composite material used in load bearing areas, especially if bruxism exists, the creep properties are of interest. The creep properties of dental composites under conditions of optimal conversion are influenced by the content and type of filler as well as the final structure of the organic matrix (63-65). The inclusion of inorganic fillers and particularly silane treated filler particles appears to reduce creep and increase stiffness (66,67). The stress/strain behavior in compression of dental composites reveals considerable differences between a material with a high filler content like P-10 (71 vol.-%) and a material with a lower inorganic filler content like the microfill material Heliomolar (KM) (Vivadent) (45 vol.-%). It has been claimed that posterior composites are brittle materials (Lloyd, 19830. However, in compression the stress/strain curve of HM reveals a yield point indicating that HM behaves more like a tough material, whereas P-10 appears to be a brittle material. On the basis of the assumption that the matrix phase breaks at the same deformation whether filled or unfilled (i.e. good adhesion between filler and matrix) a decrease in observed nominal strain with increasing quantity of fillers is predicted (68). This means a decrease in ductility with increasing quantity of inorganic particles. In general, the elastic modulus increased with increasing volume fraction of inorganic fillers- When stored in water until saturation, a decrease of elastic modulus and ultimate strength of dental composites can be observed (65).

Creep characteristics of dental composites have been determined (63-65). Uniaxial compressive creep experiments with different loads can be expressed as strain/time functions. If the Boltzmann superposition principle, which implies that strain is proportional to stress, holds, the strain/time functions can be transformed to one single creep compliance/log time function. if there is good adhesion between the silanized rigid fillers and the polymer matrix, all deformation occurs in the polymer phase (63). i.e. the true deformation of the polymer phase is much greater than the observed nominal strain. Creep results of dental composites indicate that creep properties are dependent on the volume fraction of inorganic particles. water uptake and the structure of the polymer matrix.

Creep measurements indicate increased creep after water saturation. Water absorbed by the polymer matrix may act as a plasticizer on the materials (69), and thereby increase creep. It has been reported that soaking in water slightly increases the creep of filled polyethylene. especially if the filler particles are not silanized (70). The extent to which properties of composites could be improved by optimizing interfacial bonding between filler matrix, and reported that tensile strength and water resistance varied with silanization conditions. Therefore. differences in creep increase after submersion in water could also be due to variations in bonding between filler and matrix in dental composites. Filler-matrix debonding can also be caused by hydrolytic degradation of the fillers (71,72). Leaching of inorganic ions from the fillers may adversely affect the hydrolytic stability of the bonding between the polymer and filler by the organo-siloxane layer. As expected. the creep compliance increases with decreasing content of inorganic filler. revealing that the microfilled materials have the highest creep values (63,65).

Creep results of dental composites indicate that creep properties are dependent on the volume fraction of inorganic particles, water uptake and the structure of the polymer matrix. The structure of the polymer matrix is dependent on the structure of the monomers and oligomers used and their conversion after the polymerization reactions. The conversion is also dependent on the structure of the monomers and oligomers as well as on the method for initiation of polymerization.

It is difficult to predict which mechanical properties should be emphasized for composite dental resins. A need for studies correlating various mechanical properties to clinical performance is needed, especially the establishment of optimal requirements.

2.7 Remarks

As pointed out by Dr. Leinfelder, there are no laboratory methods which reflect the wear stability in vivo. Laboratory testing methods for this purpose should include influences of environmental, oxidative, dynamic, mechanical and thermo-mechanical nature. Due to the diversity in composition, and therefore in the properties of dental composites, it is difficult to predict which factors are most important to determine for these types of materials. Presently no short-term tests, whether laboratory or clinical, are suitable for predicting the long-term clinical behavior of dental composites. It is unlikely that a clinically relevant laboratory test may be found. Thus, emphasis must be placed on clinical assessments to evaluate wear, especially methods for determining the interproximal wear.

A prediction of the long-term clinical behavior of non-microfill materials based on short-term clinical investigations with indirect evaluation may appear possible. It has been indicated by Dr. Leinfelder that the mechanism of wear for microfill materials, as well as

for some selected composites, is different from the wear of nonmicrofill materials, i.e. a localized type of distraction occurs.

The quantitative difference in wear of occlusal contact areas and contact-free areas should also be considered when predicting the long-term clinical behavior of dental composites. There has been demonstrated a threefold difference in wear between the contact and noncontact areas (73).

Poor long-term durability of dental composite resins, especially the extensive degradation/wear due to occlusal and approximal attrition in class II restorations, has resulted in several changes to improve the properties of restorative composites. Several types of fillers, reduction in size of the fillers and higher filler loading have been introduced to improve the in vivo wear properties. The hydrolytic stability of the fillers, which is closely related to the hydrolytic-stability of the bond between matrix and fillers, must be considered.

Both clinical and laboratory studies indicate that the resistance to degradation of the organic matrix is of great importance for the stability of composite resin restorations. Poor conversion in dental composites leaves a large amount of reactive methacrylate groups in the polymerized restoration. The carbon-carbon double bonds of these pendant methacrylate groups (PMG) can make the polymer matrix susceptible to degradation reactions.

The need for short term tests which are predictable for long-term performance is present. However it seems that the type of filler will have a decisive effect on the prediction, and specific tests may therefore be needed for different types of composite dental resins. Several new di- and oligo- methacrylate systems have been introduced.

However, new resins systems should be developed with the aim of achieving better conversion. Also low polymerization contraction and good mechanical and biological properties must be considered. An important factor which also determines the degree of conversion is the initiating system. Good clinical results in posterior teeth have been obtained for UV-activated composites. UV-activation is more effective for radical formation than for chemical initiation. UV-activation also yields better optimal conversion than visible light activation. It is further observed that the conversion of visible light activated materials is dependent on the spectral distribution of the light source. Furthermore, it has been demonstrated that the conversion, as well as the structure of the monomers/oligomers and type and quantity of the filler particles determine mechanical properties, in casu creep, of the polymerized composite materials.

CHAPTER 3

MATERIALS AND EXPERIMENTAL METHODS

Polymers used for restorative and other uses in dentistry cover a wide spectrum of materials from elastomeric types on the one hand (e.g., soft liners and maxillofacial materials) to rigid and hard materials on the other (e.g., filled resins and denture base resins). Because of a wide range of mechanical behavior involved, and the close relationship of this mechanical behavior to the viscoelastic properties centered around one or more glass transition phenomena, dynamic mechanical analysis (DMA) is a very valuable method to characterize dental polymeric systems. The sensitivity of the DMA (figure 1) for the detection of glass transitions is well known and many investigators have used the technique to characterize viscoelastic properties of different dental polymers. The information provided in DMA tests such as storage modulus, loss modulus and tan delta have been used to study heat cure denture resins in the past. While the storage modulus (E') represents the stiffness or rigidity of a material by defining the stress to strain ratio during elastic deformation (i.e., the proportionality constant between stress and strain), the loss modulus is associated with the energy absorbed by the resin to increase its segmental molecular vibration or translation of chain positions during dynamic deformation. Such absorption of energy for molecular segmental vibration or again translation rather than for elastic deformation results in damping of the amplitude of specimen oscillation during dynamic deformation. Tan delta, the ratio of loss modulus to storage modulus (i.e., E''/E') is also used to define damping in the system. Composites are subject to stress cycles during mastication. The composite resin should be sufficiently stiff (i.e., possess adequate

flexural modulus) without at the same time being too stiff which may make the denture

brittle and cause its premature failure during mastication. There is also potential influence

of transitions and damping effects on creep behavior of composites. For these reasons,

flexural modulus and other dynamic mechanical properties of composite resins are of

importance to ensure durability of restorations composites during their clinical service.

DMA analysis provides valuable information not only on these properties, but also on the

transitions and structural changes in the resin formulations, with differences in their

compositions.

3.1 Thermogravimertric Analysis

Thermogravimertric analysis (TGA figure 2) is one of the most widely used thermal

analysis techniques. It specifically measures the weight changes (gain or losses) in the

materials. Such analysis provides information about the material's thermal stability as well

as the material's compositional makeup. TGA operates on a null-balance principle, using a

highly sensitive transducer coupled to a taut-band suspension system to detect minute

changes in the mass of a sample. An optically actuated servo loop maintains the balance

arm in the horizontal reference (null) position by regulating the amount of the current

flowing through the transducer coil. An infrared LED light source and a pair of

photosensitive diodes detect movement of the beam. An optically actuated servo loop

maintains the balance arm in the horizontal reference (null) position by regulating the

amount of current flowing through the transducer coil. An infrared LED light source and a

pair of photosensitive diodes detect movement of the beam. A flag at the top of the

balance arm controls the amount of the light reaching each photosensor. When weight is

lost or gained, the beam becomes unbalanced, causing the light to strike the photodiodes unequally. The unbalanced signal is fed into the control program, where it is zeroed. This changes the amount of the current supplied to the meter movement, causing the balance to rotate back to its null (zero) position. the amount of current is directly proportional to the change in the mass of the sample.

3.2 Thermo Mechanical Analysis

Thermo mechanical analysis (TMA figure 3) measures linear or volumetric changes in the dimensions of a sample as a function of time, temperature and force (thermal expansion). The value of thermomechanical analysis stems from its ability to measure linear or volumetric changes in the samples as they are subjected to heat and mechanical distortion. The heart of the TMA is a movable -core linear variable differential transformer (LVDT) whose output is proportional to the linear variable displacement of the core caused by changes in the sample dimensions. Force is applied by an electromechanical coil, and the heat by a precisely controlled low - mass furnace. The sample chamber, located in the core of the furnace, also has provision for cooling and atmosphere control. A thermocouple adjacent to the sample assures accurate measurement of sample temperature.

3.3 Thermal Expansion

Thermal Expansion is an important criteria to be tested for dental composites. Clinical significance of thermal expansion: (a) As the temperature fluctuates within the oral cavity, the restorative expands and contracts at different rates. (b) Fluctuations as a result of expansion and contraction will not only break bonds between the restorative materials and

tooth structure. but also result in penetration of oral fluids and debris into the margin.

When a molecular model of a solid isotropic material is considered, it is seen that the molecules are held together in a regular array by forces of electrical origin: i.e. vander walls forces. These forces are weak when compared to chemical bonds, but are strong enough to prevent dissociation of the molecules into a gaseous state. At any temperature thermal forces result in vibrating of the molecules. As the temperature increases more energy is pumped into the system, the amplitude of vibration increases and as a direct consequence the average distance between molecules increases. This leads to expansion of the body as a whole as the temperature is increased and the resultant change in any linear dimension of the solid such as length, width or thickness is called linear expansion. The amount of linear expansion of a material is found to be proportional to the original length of the material and the change in the temperature.

$$\Delta L = \alpha \; L \; \Delta T$$

Where

ΔL = the change in length

α = coefficient of linear expansion

L = original length

ΔT = change in temperature

which may be expressed in terms of

$$\alpha = \Delta L \, / \, L \; \Delta T$$

The volumetric coefficient is roughly three times the magnitude of linear coefficient over equivalent temperature ranges and both are usually expressed in the unit ppm/°c. Thermal Expansion can be measured by TMA.

Three composite systems were evaluated in this investigation and are listed in table 1 with information on their manufacturer and primary composition in the formulation. The materials were selected from popular commercial brands to include two different types of filler loading. The hybrid system i.e. pertac hybrid and conquest crystal have filler particle size 2.5μm maximum and the minimum 0.1μm. It is a universal radiopaque hybrid composite system. It contains bi-functional methacrylates. The volume portion of organic fillers is nearly about 75-80% . 28mm x 10mm x 3mm rectangular bars were fabricated from the different composites studied. All the specimens were prepared in a metal mold. The fabrication procedures followed the manufacturer's recommendations. The specimens were visible light cure. the specimens were cured from one side, then turned over and cured from the other side as well to ensure adequate curing. The specimens were cured for 40 seconds. Then the specimens were kept into a humidifier which was set to 37°C for 24 hours. Next day specimen was checked for viscoelastic properties.

The dynamic mechanical properties of the four composites studied were determined in a TA Instruments DMA model 98. The instrument was interfaced to a computer control station Thermal Analyst 2000. Instrument control was carried out by the analyst system software. Dynamic mechanical analysis was carried out in fixed frequency flexural mode (1 Hz) with a pre-optimized oscillation amplitude of O.4mm. Temperature range studied varied from -50 to 180°c using a liquid nitrogen reservoir. A sample size of N-7 was used. TA instruments software DMA version 4.2 was used for analysis, where appropriate.

TMA analysis of disc specimens (6mm dia x 3mm thick) was also carried out in the temperature range of 0 to 140°C in TA Instruments TMA model to study thermomechanical behavior in selected cases to better understand the DMA curves and

thermal expansion of composite at 37°C. Analysis was done with an expansion probe with no load to follow thermal expansion changes. In addition, TGA analysis was performed in the temperature range of 25 to 800°C and this was used to characterize weight change dependence on temperature and filler contents. TMA and TGA analysis were also conducted using Thermal Analyst 2000. TA Instruments TMA software version 4.0 and the general analysis utility version 4.0 were used for the TMA and TGA analysis, respectively, where appropriate.
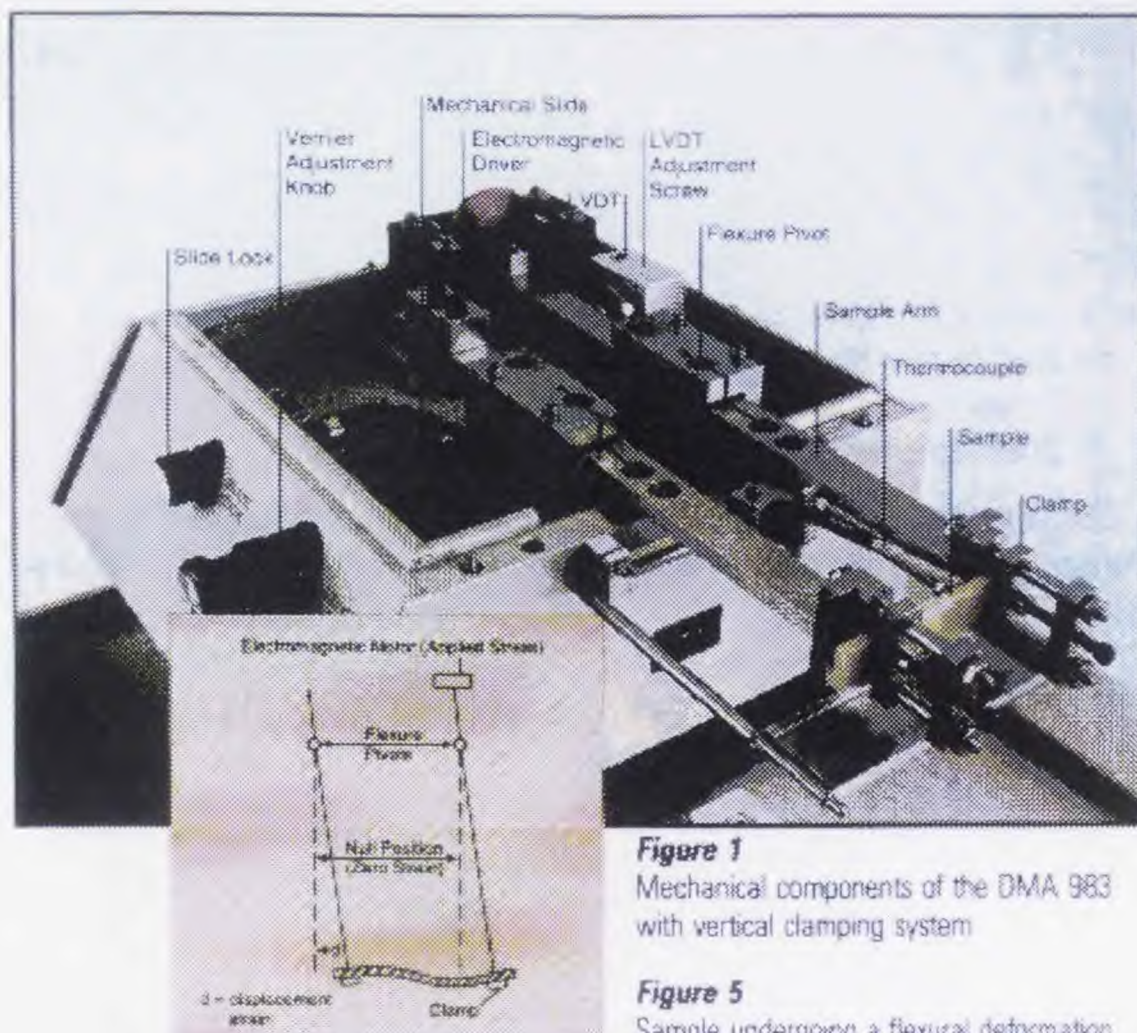
Figure 1
Mechanical components of the DMA 983
with vertical clamping system

Figure 5
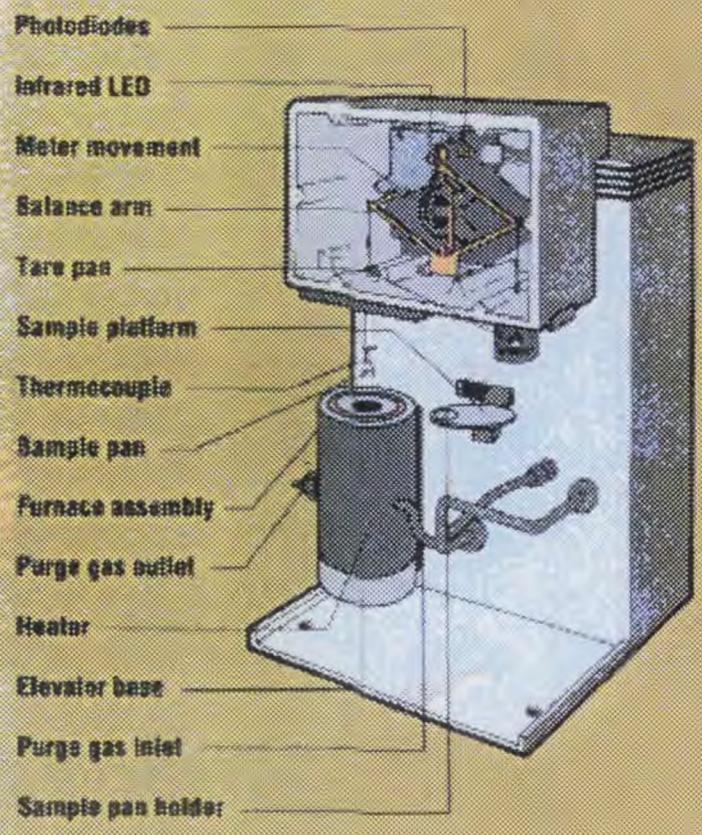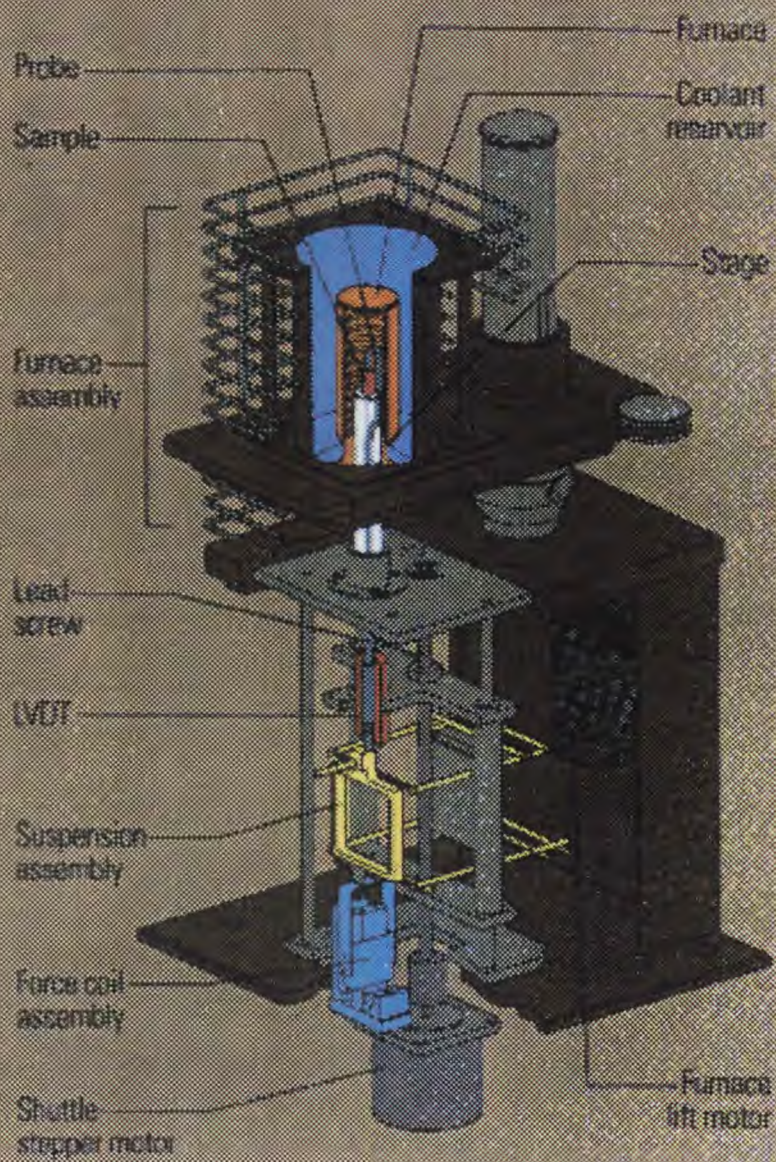Sample undergoing a flexural deformation

Figure 1.
TGA Schematic

Photodiodes

Infrared LED

Meter movement

Balance arm

Tare pan

Sample platform

Thermocouple

Sample pan

Furnace assembly

Purge gas outlet

Heater

Elevator base

Purge gas inlet

Sample pan holder

Figure 2
TMA 2940's electromechanical system.

Probe

Sample

Furnace
assembly

Lead
screw

LVDT

Suspension
assembly

Force coil
assembly

Shuttle
stepper motor

Furnace

Coolant
reservoir

Stage

Furnace
lift motor

CHAPTER 4

RESULTS

The results of the different investigated properties are listed in table 1. The four different composites are divided into two categories: Hybrid and Microfill composites. Examples of Hybrid composites are - Pertac Hybrid, conquest crystal and Prodigy. Visiofil is a microfill system. All four composites were analyzed using DMA, TMA, and TGA. Figures 4 to 7 show plots of DMA. Figures 8 to 11 show plots of TMA. And from figures 12 to 20 show TGA cures. Composites were analyzed in TGA using derivative curves also. From the table it has been observed that storage modulus is in the range of 2 to 9 Gpa. Loss modulus is in the range of 268 to 811 Mpa. and Tan Delta was in the range of 0.06 to 0.012. Thermal expansion coefficient from TMA was in the range of 46 to 77 $\mu m/m^{\circ}C$. From the TGA curves for hybrid and microfill composites major breakdown temperature was observed in the range of 280 to 400°C.

4.1 Discussion of Results

Composites have been available to the dental profession for many years. The results of this study reveal interesting differences between hybrid and microfill composites. The composites were analyzed for dynamic flexural behavior, thermal dimensional expansion including thermal expansion coefficient differences and thermal breakdown characteristics between the different composites studied.

DMA(model 983) was used to analyze viscoelastic properties of composites. Fix frequency mode was used from the table 1 and from fig. 4 to 7, we can observe that hybrid

33

composite has higher elastic modulus (E') than that of microfill composite. And again loss modulus is also higher in case of hybrid than that of microfill system. Higher loss modulus and higher storage modulus is due to higher filler content. Due to high storage modulus, hybrid composites are more rigid than that of microfill system. Damping characteristics observed as higher or lower delta peak also show interesting differences. Visio (microfill) composite has higher value than two other hybrid composites, Pertac and Conquest. Tan Delta is a ratio of loss modulus (E'') to storage modulus(E'). It is a very useful parameter, because it expresses in a single number, the relative importance of viscous and elastic processes in a material. From the table we can see that at 37°C , microfill system has higher tan delta value than that of hybrid system. We can also observe that prodigy has higher tan delta value than conquest crystal and pertac hybrid. This can only be explained by, different particle sizes used by different manufactures (Kerr, Jeneric pentron, ESPE). Conquest crystal and pertac hybrid has tan delta values of 0.08 and 0.06 respectively, which are lower than the value of visiofill (0.13). Material with low tan delta is elastic, lively, bouncy, resilient, springy and often consider to be of high quality and applications requiring stable elastic behavior.

Temperatures corresponding to tan delta peaks were also evaluated for each composites. Microfill composites has lower tan delta peak temperature than that of hybrid composites. From the table 1 we can notice that viscofil has tan delta peak temperature of 58°C , but hybrid composites has tan delta peak temperature values in the range of 63 to 104°C , which is higher. Loss modulus peak (obtained at low frequencies) is often considered as a better glass transition temperature (Tg) than tan delta peak. But here in this study all the composites were analyzed at room temperature. so glass transition peak

at low frequency is not available. Tan delta also resembles glass transition temperature(Tg), but it always occurs at approximately 5 to 15°C higher. Higher temperature peak observed may result from two possible causes : 1) The higher degree of cross linking in composites. 2) And the presence of high filler content (about 65 to 80%). The above two factors also influences the dimensional and thermal expansion coefficient values observed in TMA analysis, because both filler concentration and cross linking would tend to lower the thermal expansion coefficient and overall dimensional changes. Visiofill shows a relatively higher (77 μm/m°C) thermal expansion coefficient than all other hybrid composites (which is in the range of 46 to 72 μm/m°C). Tooth enamel has lower thermal expansion coefficient (14x10 -6 m/m°C). From the table 1 we can observed that, prodigy, conquest crystal, and pertac hybrid has lower thermal expansion coefficient. Because of these thermal expansion matches with the tooth enamel thermal expansion. Thus there will be less microleakage. TMA curves (Figures 8 to 11) were also analyzed for dimensional change over a range of 25°C to 60°C. Hybrid composites has % dimensional change in the range of 0.18 to 0.25 %, microfill composites has 0.28% dimensional change. This is because of hybrid composites has higher filler content than microfill composites. Therefore microfill has less dimensional stability than hybrid composites.

Furthermore, TGA curves were analyzed for filler contents and onset temperatures of breakdown. From TGA curves it has been observed that visiofill has approximately 60% filler content and pertac hybrid has filler content of about 80%. Furthermore, for TGA curves figures 12 to 15, it was noticed that visiofill and pertac hybrid shows two onset temperature breakdown, it is observed that only one set point is exists for conquest
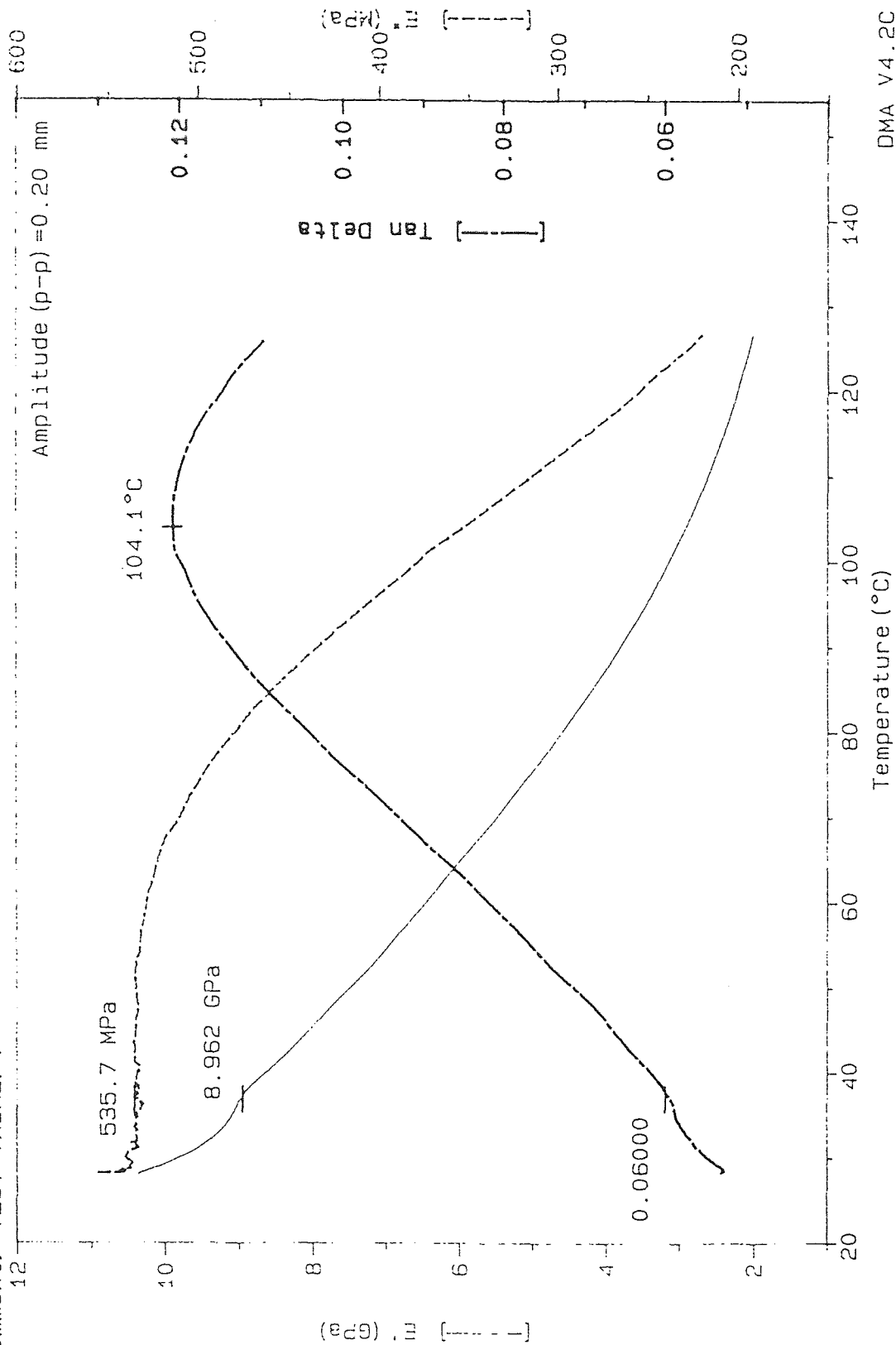
crystal and prodigy. For pertac hybrid and visiofill initiation of onset breakdown temperature has higher temperature (around 430°C) than that of prodigy and conquest crystal (360 and 311°C respectively). From derivative curves (figure 16 to figure 19) it was noticed that visiofill and pertac hybrid has three temperature breakdown regions. One at lower temperature (180°C for visiofill and 200°C for pertac hybrid), which could be because of presence of plasticezers and major temperature breakdown at around 400°C for both pertac hybrid and visiofill. This can only be explained by the similarity of resin used by manufacturer (ESPE) in formulating visiofil and pertac hybrid composites. The basic resin used in both the composite is same (Bis (meta) acylates). The high temperature breakdown was observed at around 400°C. The appearance of two decomposition peaks at the higher temperatures may indicate heterogeneity involving two phases or domains of composition or cross linking difference. The other two composites has only one major breakdown, for prodigy at 311°C and 360°C for conquest crystal.

Table 1 Properties of Hybride and Microfill Composites

Properties	Hybrid Composites			Microfill Composite
	Prodigy	Conquest Crystal	Pertac Hybrid	Visiofil
At 37°C				
Storage Modulus(E′), Gpa	6.429	6.333	8.962	2.114
Loss Modulus (E″), Mpa	811.4	556.7	535.7	268.6
Tan delta	0.127	0.089	0.060	0.127
Tan delta peak (°C)	62.19	71.43	104.1	57.78
Thermal Expansion Coefficient, $\mu m/m°C$	46.4	53.2	72.6	77
Dimensional Change (%) Range - (25 - 60 °c)	0.186	0.206	0.254	0.284
Onset temperature (°C) (major breakdown)	298	279	397	400
Filler content (%)	76	66	79	60
Temperature of peak decomposition				
Low temperature (°C)	-	-	201	179
Major (°C)	360	311	434	424
High temperature (°C)	-	-	546	541

Sample : PERTAC HYBRID
Size   : 24.4500 x 9.7400 x 2.0200 mm
Method : FFREQ
Comment: TEST-TRIAL: 4

File   : C:DPERTAC.005
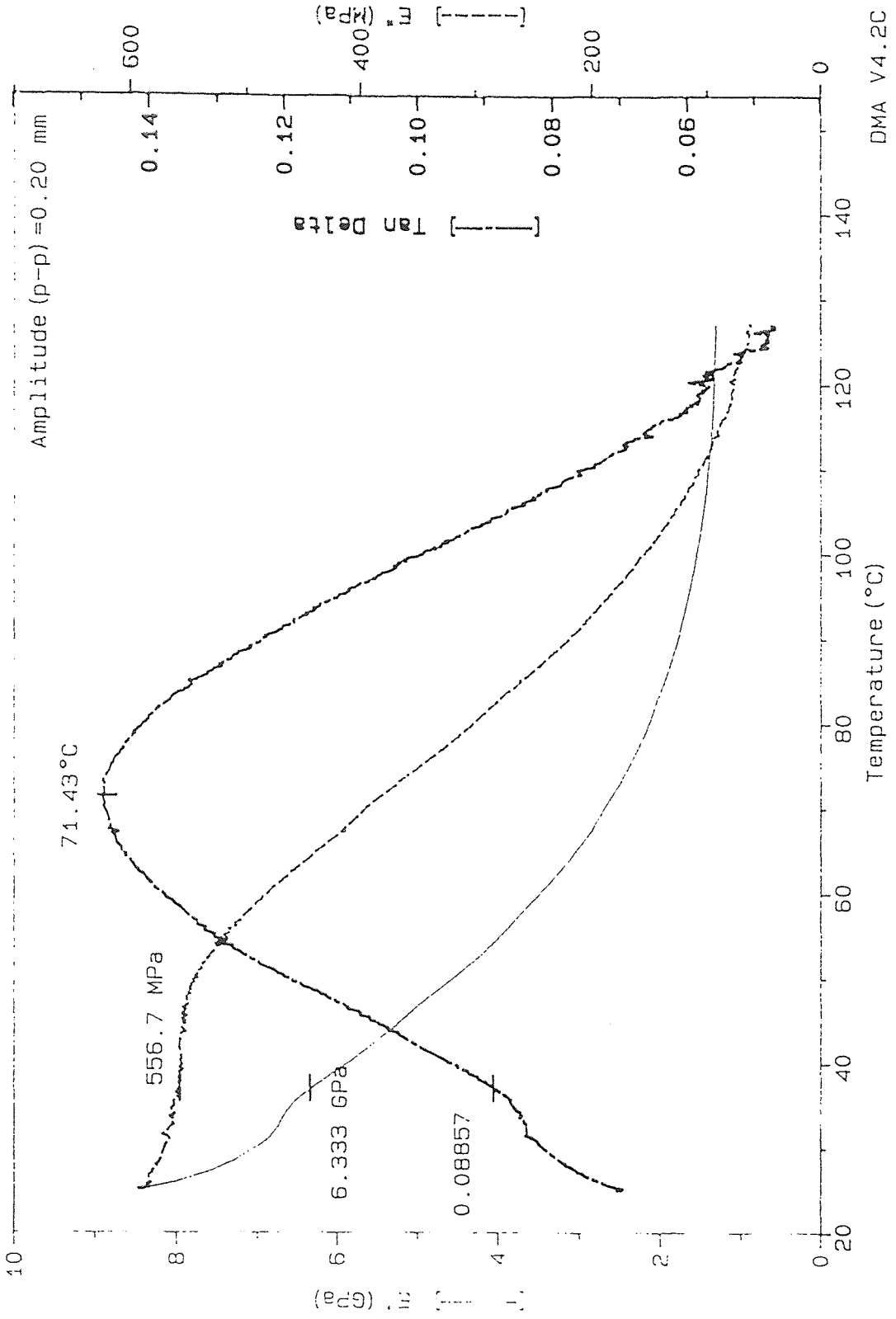Operator: PAREKH
Run Date: 16-Jan-95 19:36

DMA

Amplitude (p-p) =0.20 mm

Tan Delta [-------]

E" (kPa) [--------]

535.7 MPa

8.962 GPa

104.1°C

0.06000

Temperature (°C)

E' (GPa) [--------]

DMA V4.2C

DMA V4.2C

Sample : CONQUEST-CRYSTAL#510932-(2)
Size   : 28.8000 x 9.7000 x 2.2100 mm
Method : FFREQ
Comment: THESIS-HYBRIDE

File   : C:DCONQUEST.00B
Operator: PAREKH
Run Date: 12-Apr-95 12:08

Amplitude (p-p)=0.20 mm
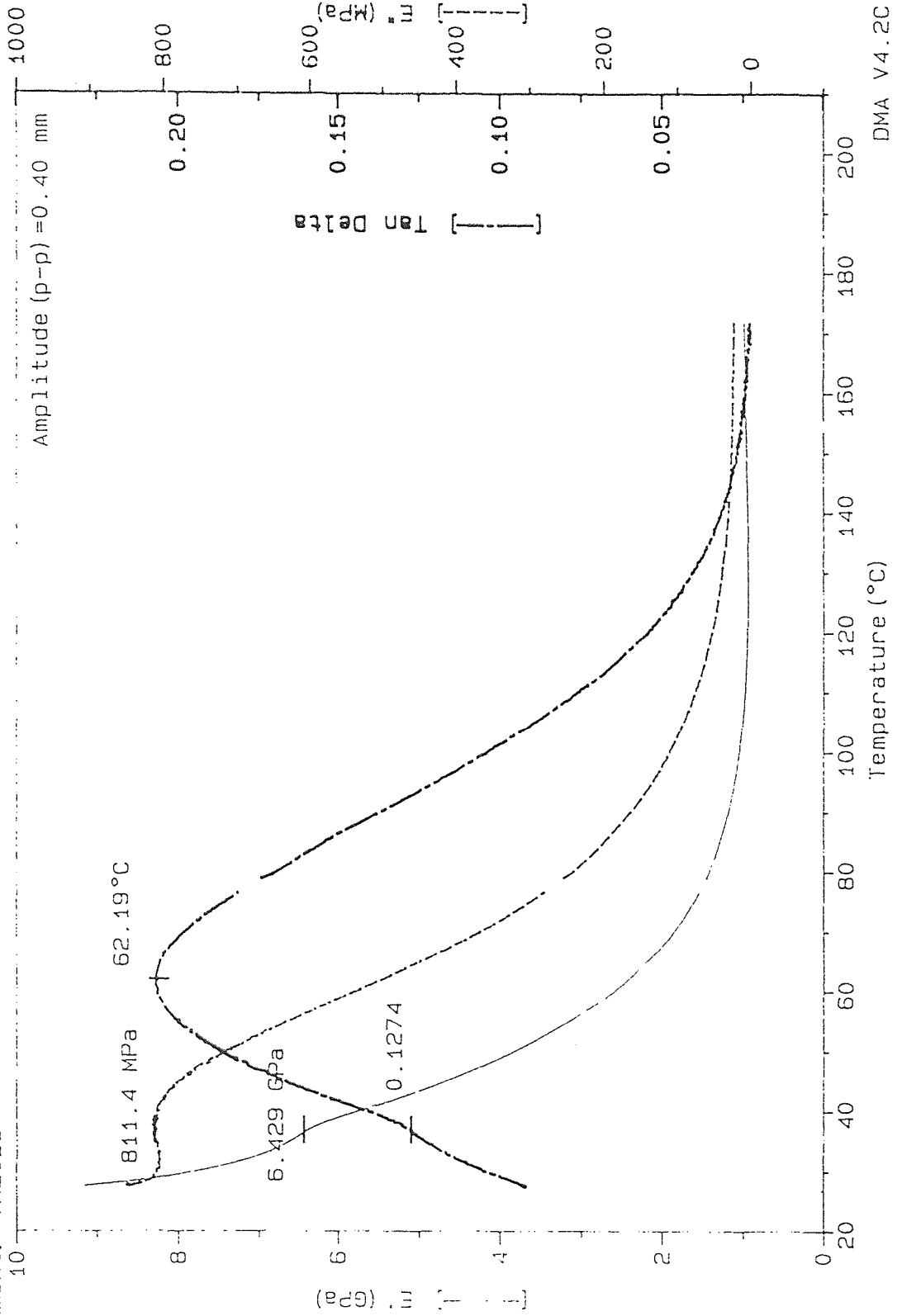
Sample : VISIO FILL NO-2
Size   : 28.5800 × 9.7400 × 2.2600 mm
Method : FFREQ
Comment: THESIS—

File : C:DVISIO.002
Operator: PAREKH
Run Date: 28-Apr-95 09:09

DMA

DMA V4.2C

Amplitude (p-p) =0.40 mm

57.78°C

0.1265

268.6 MPa

2.114 GPa

TMA
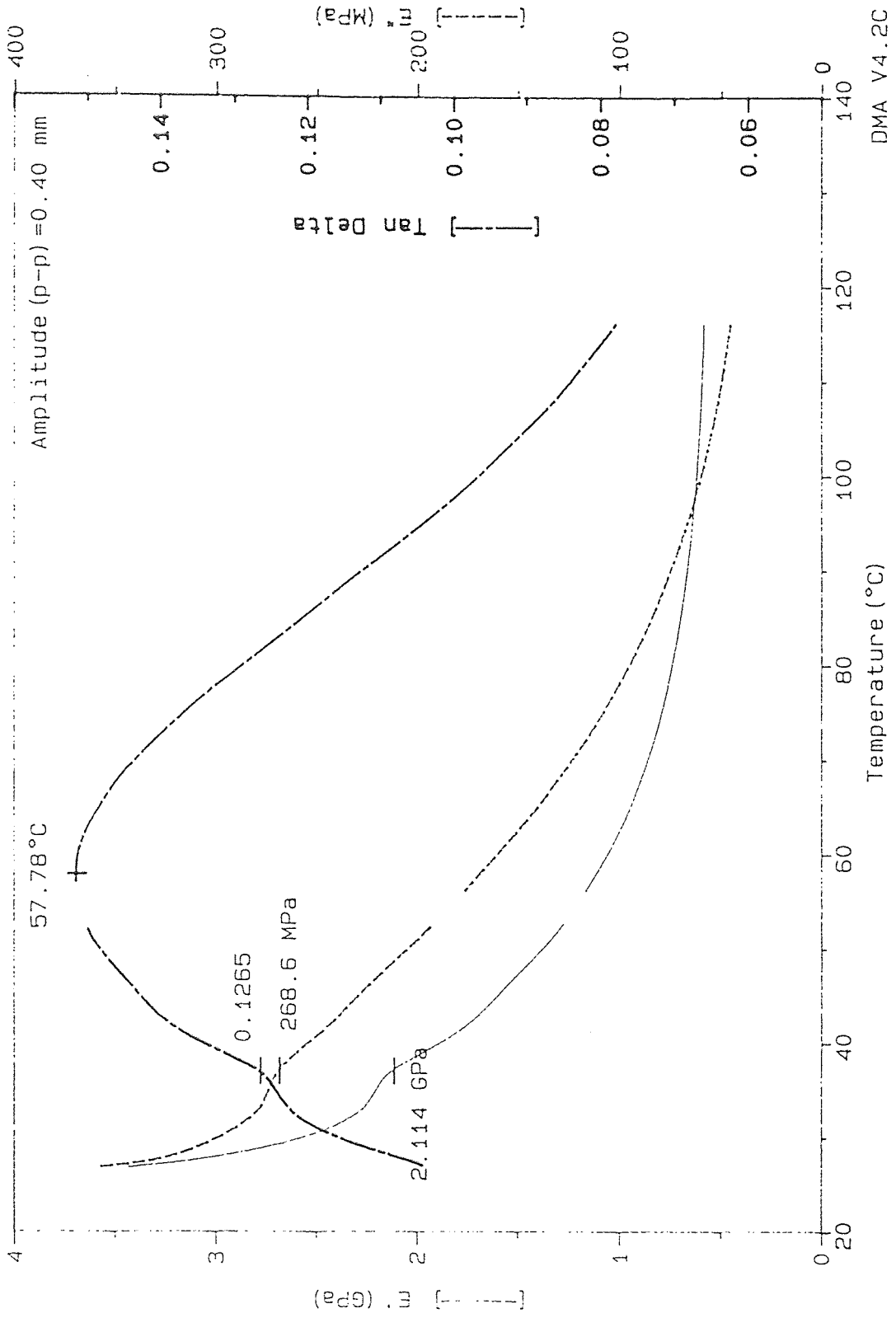
Sample: PERTAC
Size: 3.4300 mm
Method: TMA (3°C TO 200°C)
Comment: TMA 3°/MIN TO 180°C

File: C:MPERT.003
Operator: MINU
Run Date: 2-Aug-95   10:09

25.00°C

37.00°C
α~72.6μm/m°C

60.00°C
0.254%

Temperature (°C)

Dimension Change (μm)

TMA V4.0B DuPont 2000

TMA
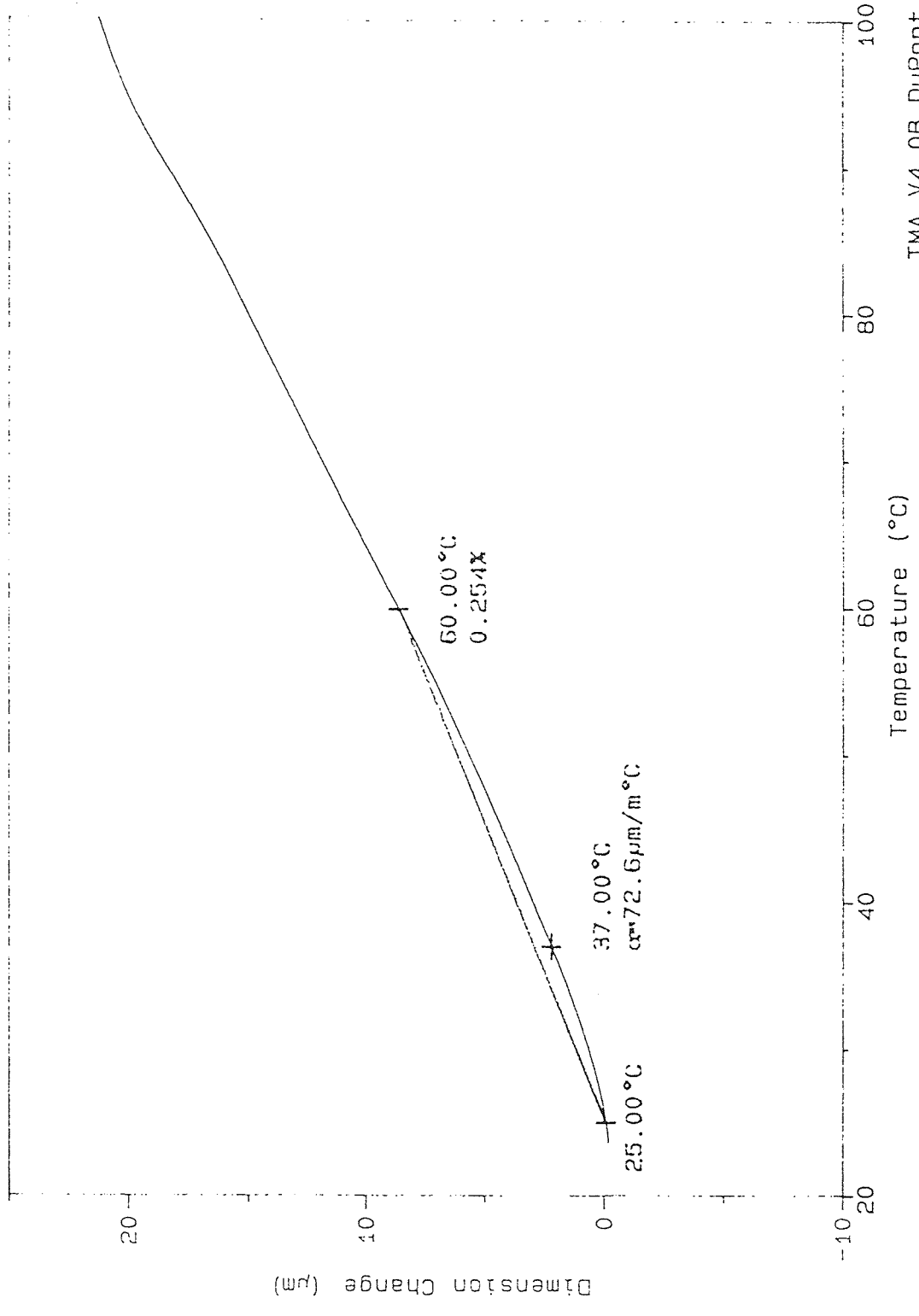
Sample: CONQUEST CRYSTAL
Size: 3.8000 mm
Method: TMA (3°C/MIN TO 200°C)
Comment: TMA 3°C/MIN TO 180°C,

File: C:MCONQ.003
Operator: MINU
Run Date: 9-Aug-95 23:18

60.00°C
0.206%

37.00°C
α=53.2μm/m°C

25.00°C

Dimension Change (μm)

Temperature (°C)

TMA V4.0B DuPont 2000

TMA
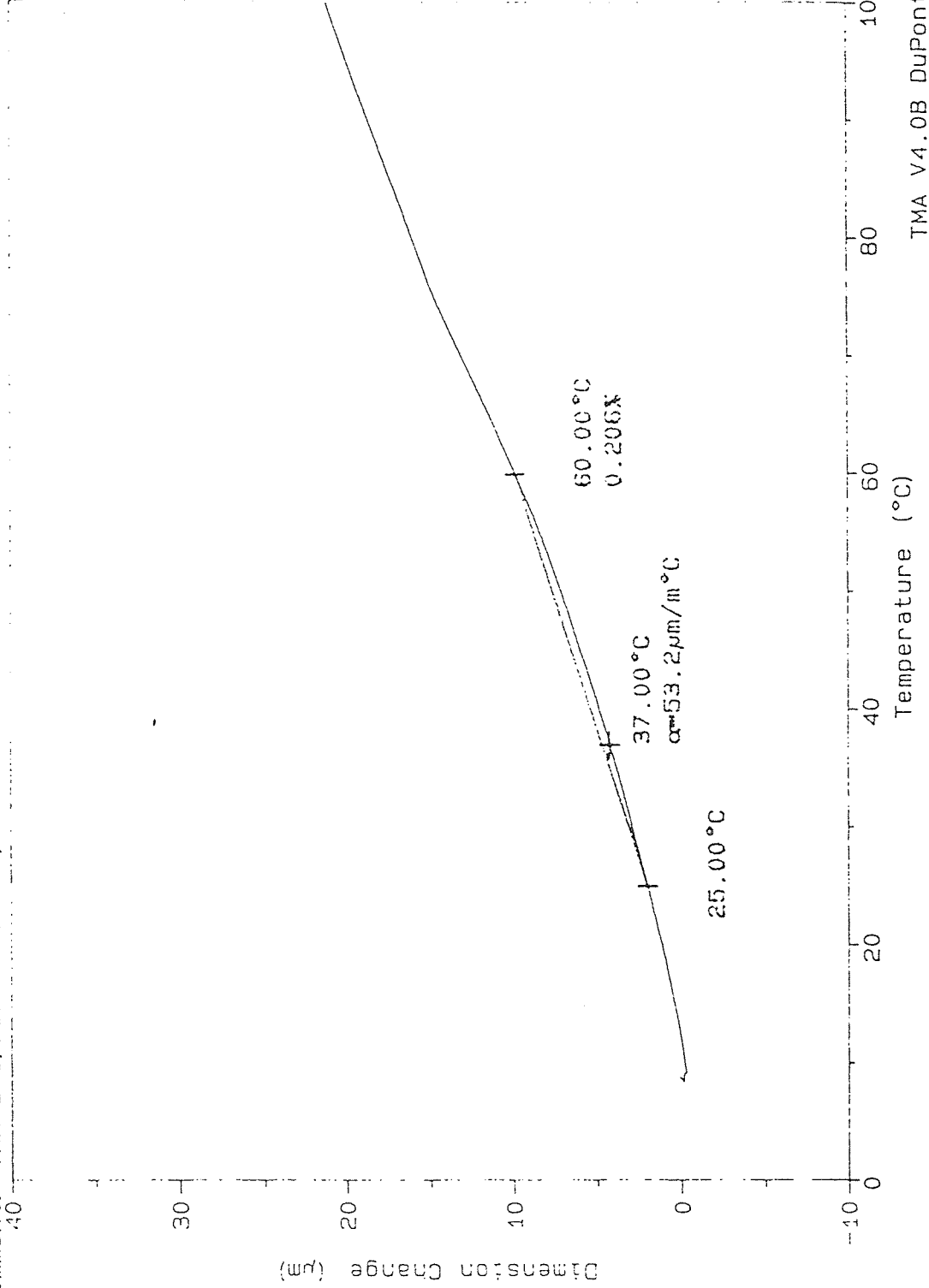
Sample: PRODIGY
Size: 3.4700 mm
Method: TMA (3°C TO 200°C)
Comment: TMA 3°/MIN TO 180°C.
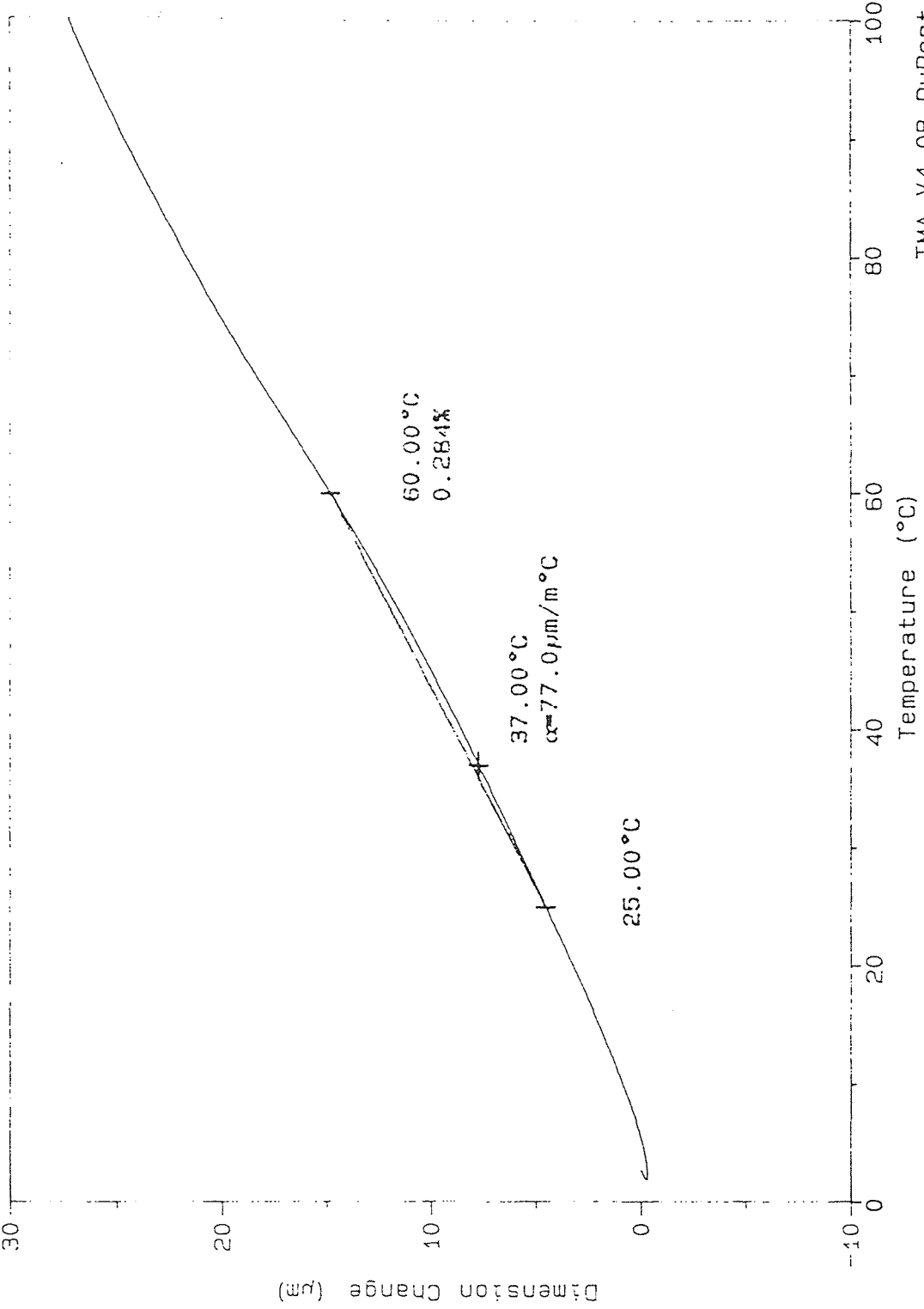
File: C:MPRO.001
Operator: MINU
Run Date: 7-Aug-95   03:50

60.00°C
0.186%

37.00°C
cr=46.4μm/m°C

25.00°C

Dimension Change (μm)

Temperature (°C)

TMA V4.0B DuPont 2000

TMA
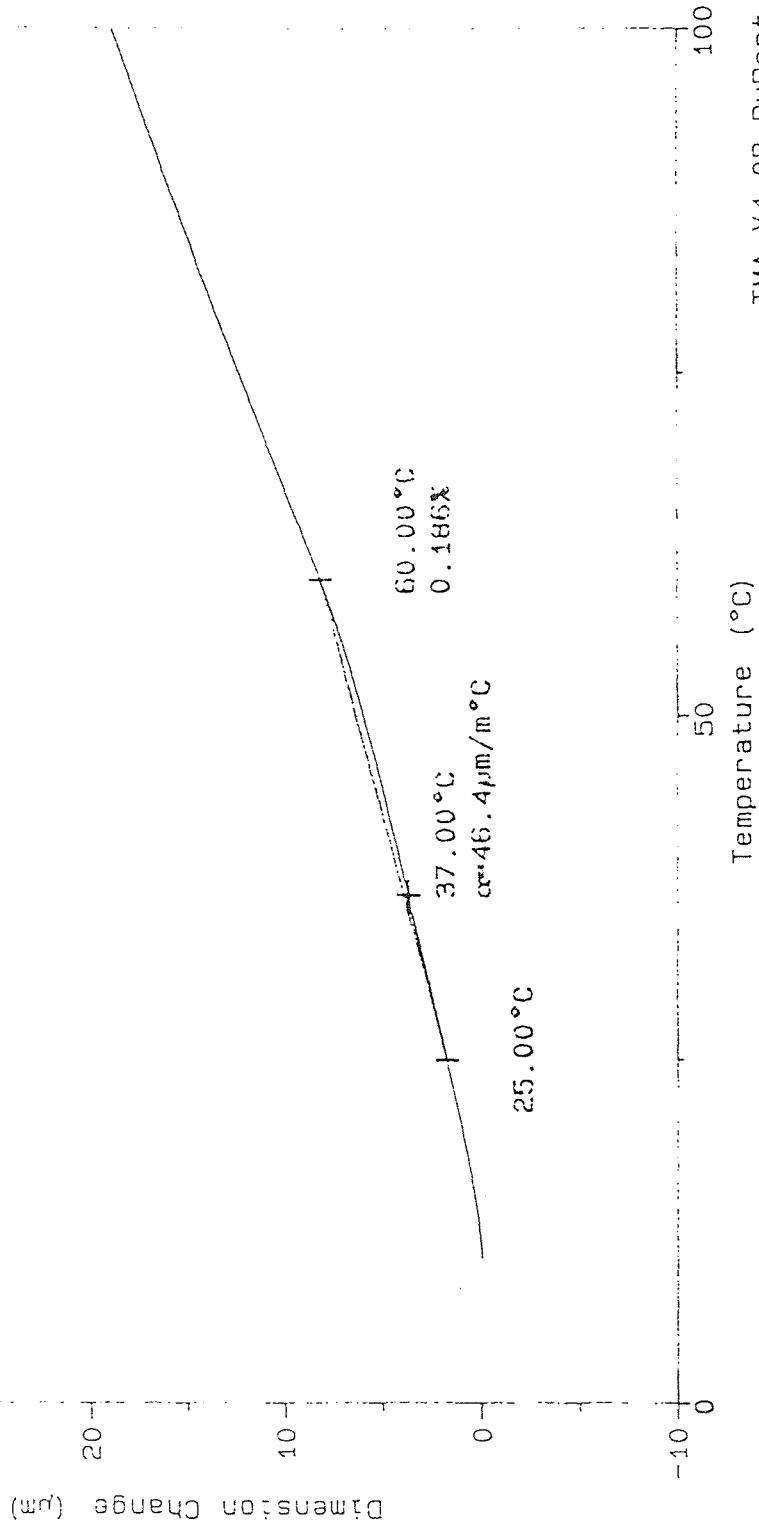
Sample: VISIO
Size: 3.6300 mm
Method: TMA (3°C/MIN TO 200°C)
Comment: TMA 3°C/MIN TO 180°C

File: C:MVISI.003
Operator: MINU
Run Date: 14-Aug-95 13:16

25.00°C

37.00°C
α=77.0μm/m°C

60.00°C
0.284%

Dimension Change (μm)

Temperature (°C)

TMA V4.0B DuPont 2000

TGA

Sample: PERTAC
Size: 5.9170 mg
Method: TGA
Comment: 25°C/MIN. TO 800.00°C

400.78°C

78.57%

Weight (%)

105
100
95
90
85
80
75

0   200   400   600

Temperature (°C)

General V4.0D DuPont 2000

TGA

Sample: CONQUEST
Size: 4.9540 mg
Method: TGA
Comment: TGA 25°/MIN TO 800°C

File: C:TCON.001
Operator: MINU
Run Date: 4-Aug-95   06:43

278.87°C

65.50%

Temperature (°C)

Weight (%)

General V4.0D DuPont 2000

TGA

Sample: PRODIGY
Size: 6.3320 mg
Method: TGA
Comment: TGA 25°/MIN TO 800°C

File: C:TPROD.002
Operator: MINU
Run Date: 4-Aug-95 04:03

298.29°C

76.05%

Weight (%)

Temperature (°C)

General V4.0D DuPont 2000

TGA

Sample: VISIO.
Size: 3.7550 mg
Method: TGA
Comment: 25°C/MINUTE, TGA

File: C:TVISIO.OOA
Operator: MINU
Run Date: 26-Jul-95 14:48

397.05°C

59.91%

Temperature (°C)

Weight (%)

General V4.0D DuPont 2000

TGA

Sample: PERTAC
Size: 5.9170 mg
Method: TGA
Comment: 25°C/MIN. TO 800.00°C

File: C: TPERT.OOA
Operator: MINU
Run Date: 31-Jul-95 05:05
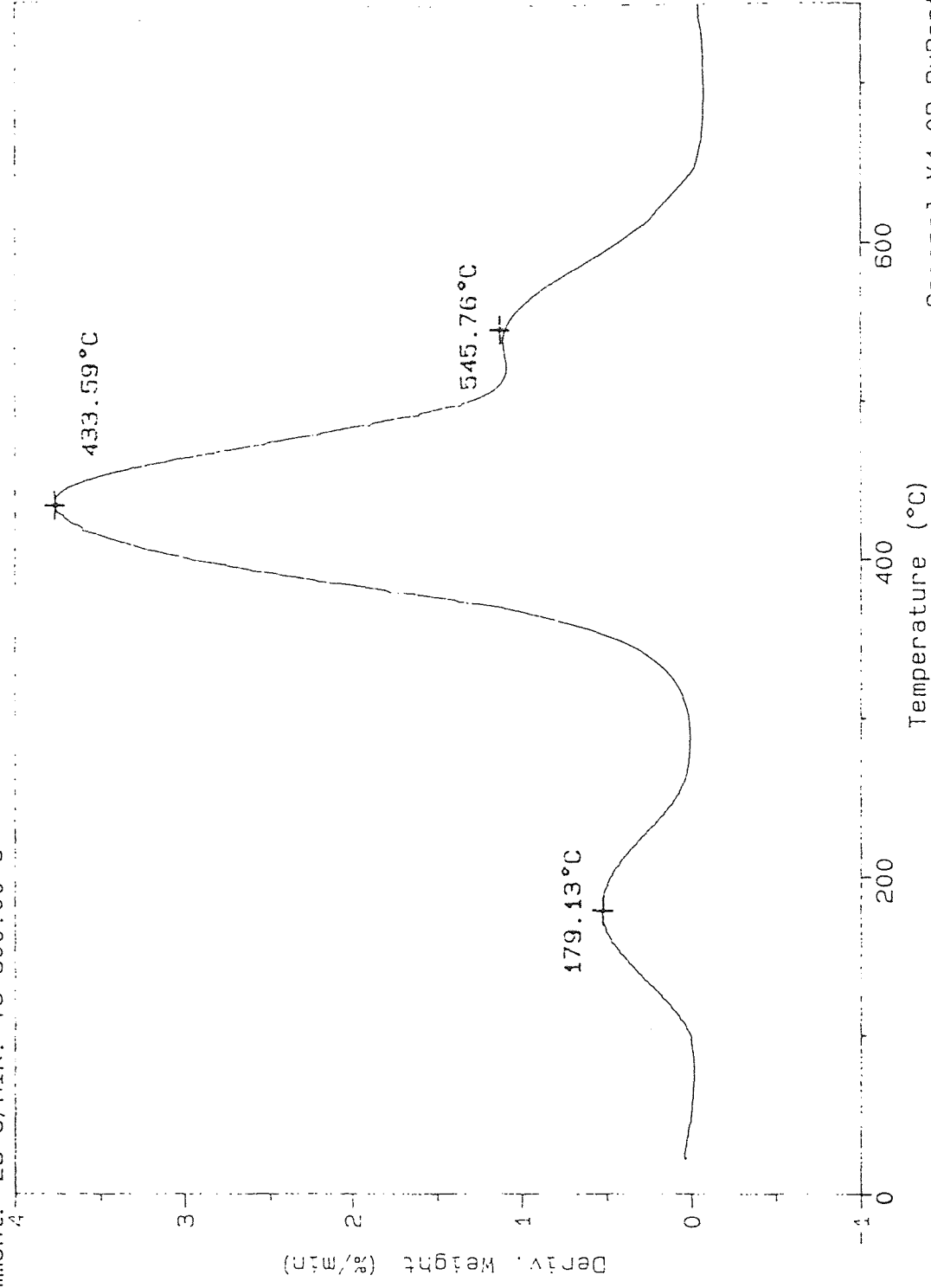
433.59°C

545.76°C

179.13°C

Deriv. Weight (%/min)

Temperature (°C)

General V4.0D DuPont 2000

TGA

Sample: VISIO.
Size: 3.7550 mg
Method: TGA
Comment: 25°C/MINUTE, TGA

File: C:TVISIO.00A
Operator: MINU
Run Date: 26-Jul-95 14:48

200.89°C

423.55°C

540.74°C

Temperature (°C)

Deriv. Weight (%/min)

General V4.0D DuPont 2000

TGA

Sample: CONQUEST
Size: 4.9540 mg
Method: TGA
Comment: TGA 25°/MIN TO 800°C

File: C: TCON.001
Operator: MINU
Run Date: 4-Aug-95   06:43

311.38°C

General V4.0D DuPont 2000

Temperature (°C)

Deriv. Weight (%/min)

53

TGA

Sample: PRODIGY
Size: 6.3320 mg
Method: TGA
Comment: TGA 25°/MIN TO 800°C

File: C: TPROD.002
Operator: MINU
Run Date: 4-Aug-95 04: 03

359.93°C

Deriv. Weight (%/min)

Temperature (°C)

General V4.0D DuPont 2000
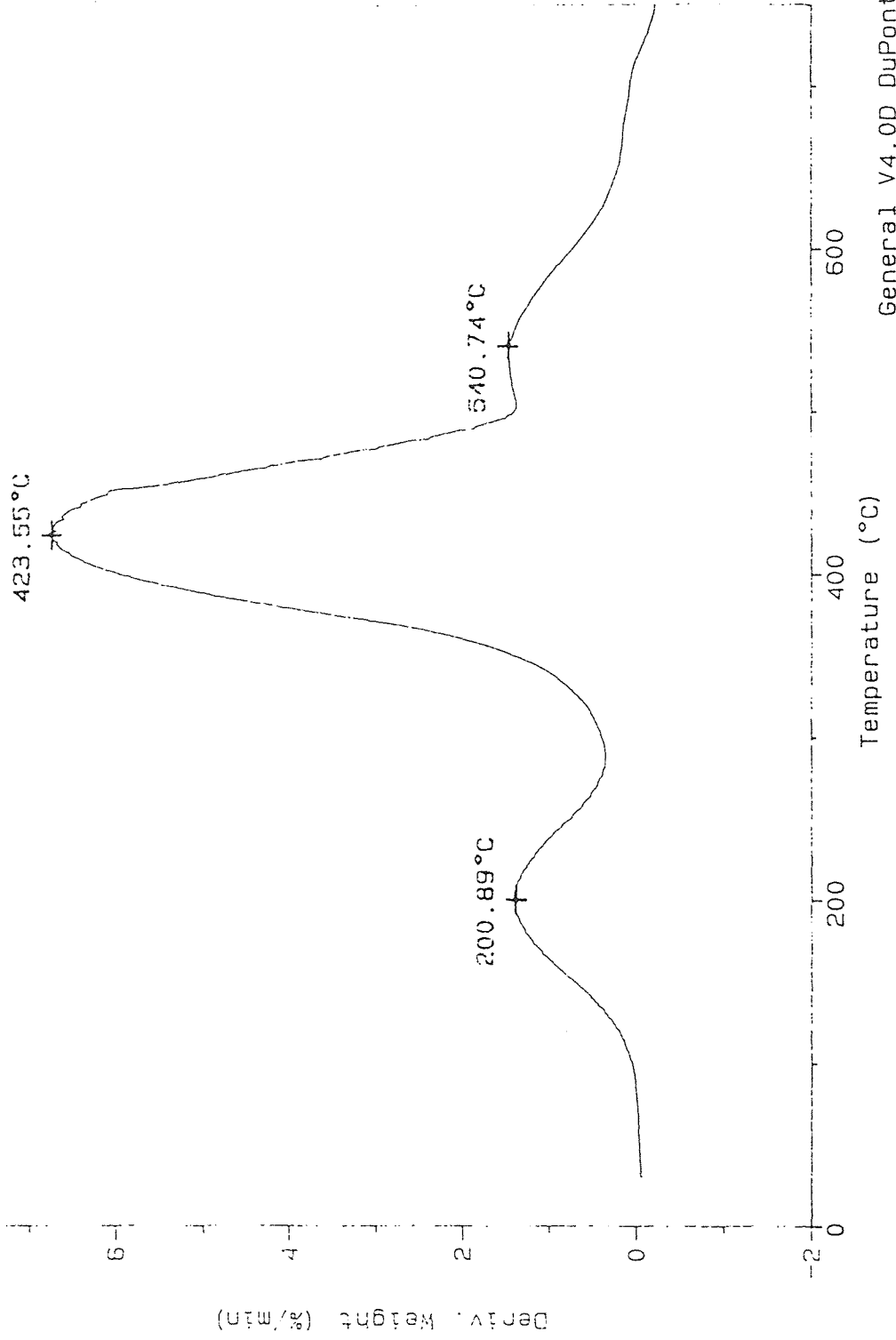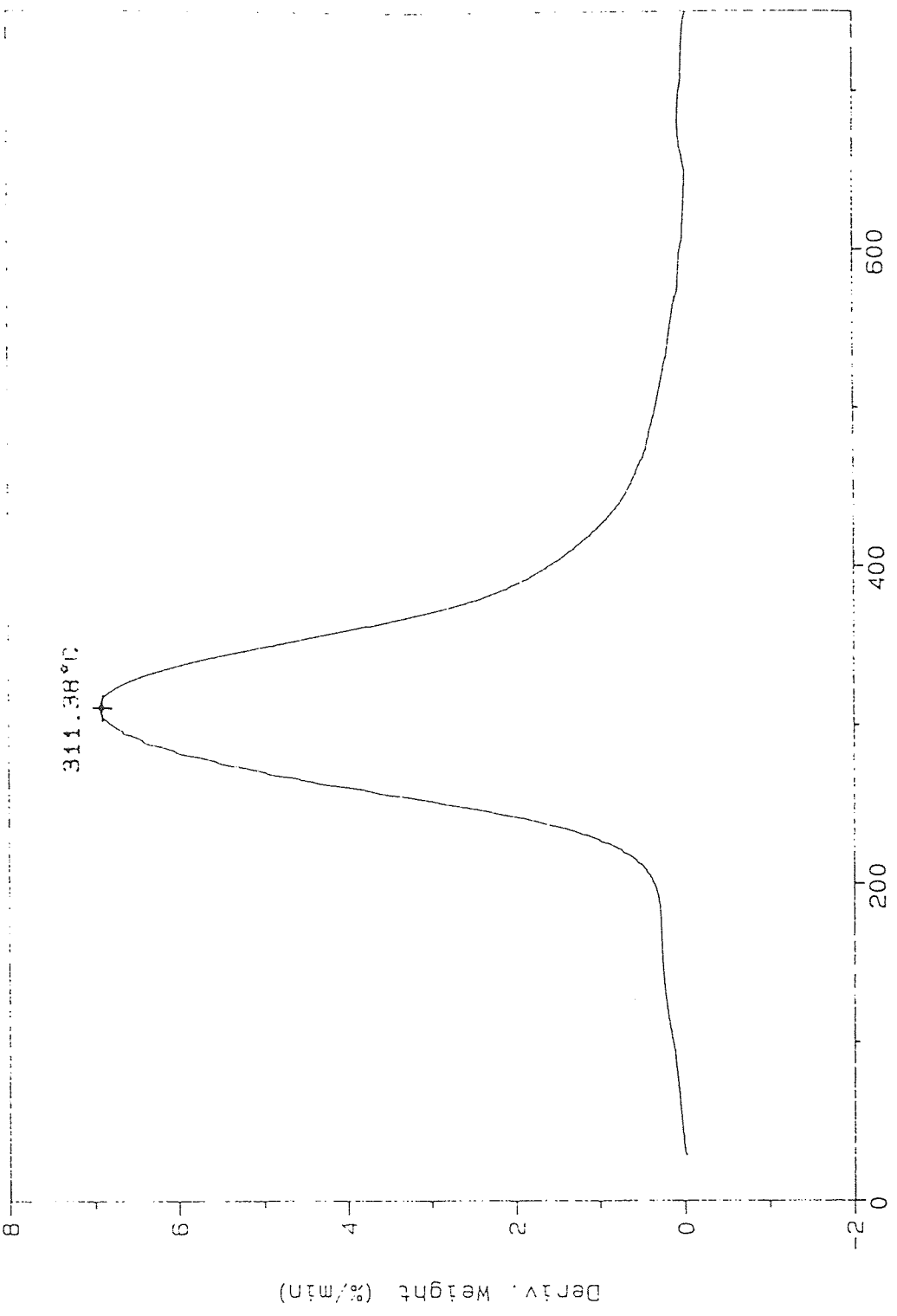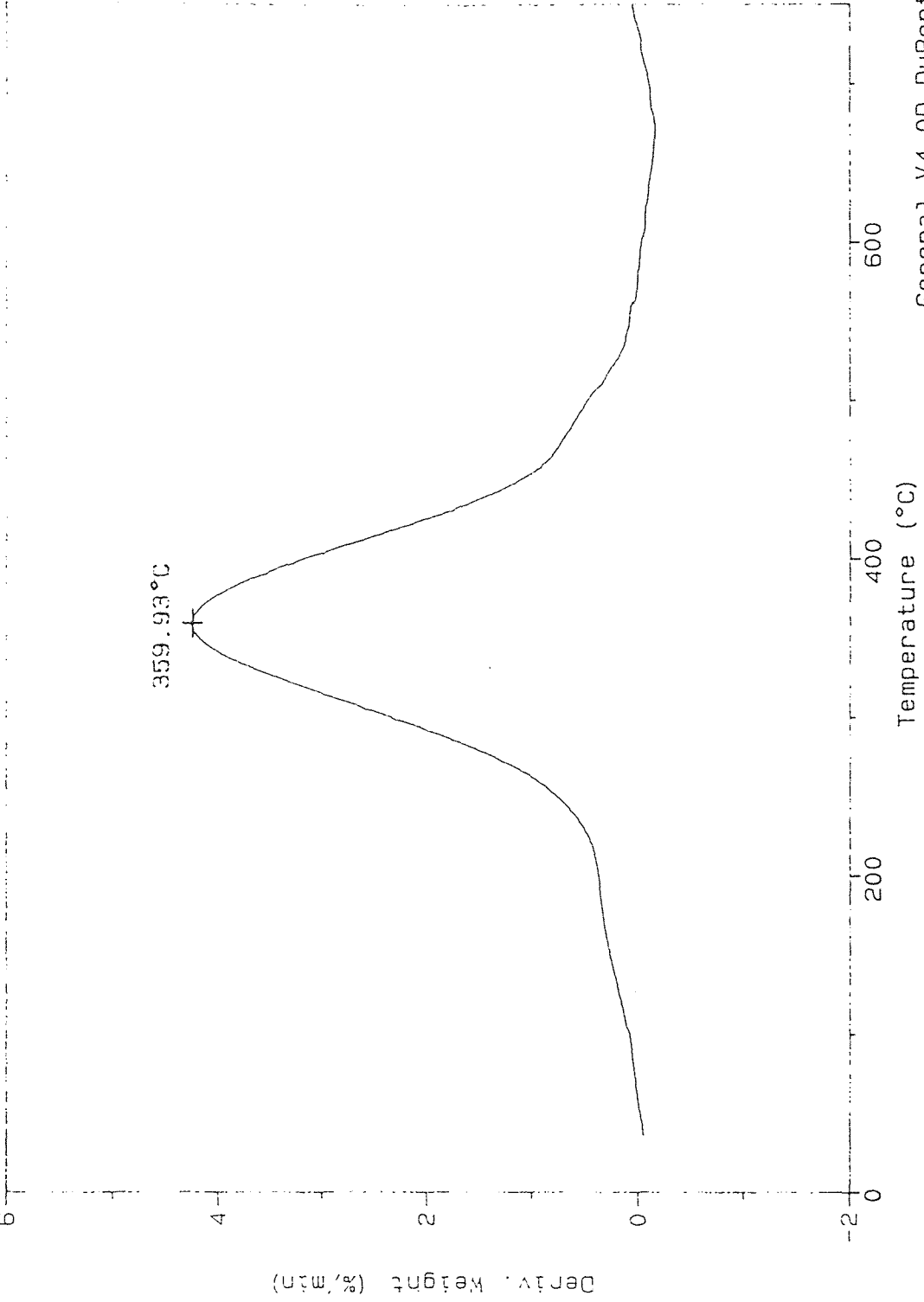
CHAPTER 5

CONCLUSION

Different restorations using composites require different properties. Class I and class II restorations (i.e., those on the occlusal or chewing surfaces) require higher rigidity and higher wear resistance. Hybrid composites are ideal for such use. On the other hand, class V restorations on the proximal surfaces of tooth are not subject to wear by chewing. However, they are subjected to flexural fatigue. These restorations should flex with tooth function to avoid brittle failure. Microfill composites are ideal for these purpose as it has lower modulus of elasticity. Microfill composites are having very fine filler size (0.4 μm). Thus they gives high palatability. Hybrid composite has higher filler content than microfill composites. Therefore, storage modulus and loss modulus are higher for hybrid composites than microfill composites. Tan delta is higher for microfill than hybrid composites. Furthermore, thermal expansion coefficient at 37 °C (mouth temperature) for microfill is higher than hybrid composites.

Thus hybrid composites are ideal for Class I and class II restorations and Microfill composites are ideal for class V restorations on the proximal surfaces of tooth.

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