Review

Titanium in Dentistry: Development and Research in the U.S.A.

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Titanium has received a great deal of attention from dental researchers and clinicians. In the United States, the history of the application of titanium as a biomaterial started as early as 1940 when experiments with titanium implants were done using laboratory animals. It appears that in 1959, two American researchers were the first to suggest casting titanium to fabricate implant posts. The use of cast titanium for dental prostheses in the U.S. was first reported in 1977. The present survey revealed that during the last decade, the number of research projects on the applications of titanium to dentistry that were presented at annual IADR/AADR meetings has steadily increased. The majority of these presentations have been made by American researchers, and 59% of these were related to dental implantology. The numbers of reports on casting and prosthodontic applications have steadily increased, but they are still more limited. The purpose of this review was to provide information on the development and the current status of research on titanium in dentistry in the U.S.

Key words: Titanium, Casting, Implant

INTRODUCTION

During the recent decade, titanium has drawn a great deal of attention from researchers in dental biomaterials and clinicians because of its excellent biocompatibility and acceptable chemical, physical and mechanical properties as well as the remarkable improvements that have been made in all phases of casting technology. The purpose of this review is to summarize the development and the current status of research on titanium in dentistry in the United States. This report was originally presented at the Third International Symposium on Titanium in Dentistry held in Leura, Australia, August, 1995, and has been updated and revised since the original presentation.

The history of the use of titanium in the U.S. as a biomedical material began about 50 years ago¹⁾. In 1940, Bothe *et al.*²⁾ implanted titanium in laboratory animals along with other metallic materials which were used for surgical applications. They reported that titanium was well tolerated, apparently due to its excellent corrosion resistance; this was later confirmed in biological fluids by Clarke and Hickman³⁾. Beder and Ploger⁴⁾ described the use of titanium for intraoral implants using dogs. They suggested making titanium disc implants by casting. For a long time, it was commonplace for custom-made bone implants to be made of cast cobalt-chromium, whereas it was preferred that prefabricated devices be made of titanium. In the U.S., Linkow⁵⁾ is the pioneer in developing milled titanium bladevent implants, which were introduced in the early 1970's. Titanium and its alloys have gradually been accepted in the U.S. for various dental and orthopedic uses.

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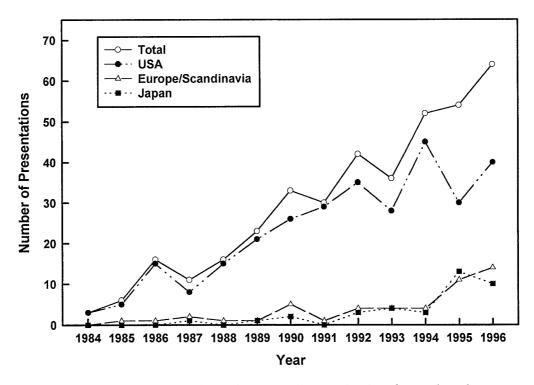


Fig. 1 Changes in the numbers of presentations on titanium from selected countries at AADR and AADR/IADR meetings (1984-1996).

Recent trends in research on the application of titanium to dentistry in the United States are apparent from the numbers of presentations related to titanium that have been given at the annual meetings of the American Association for Dental Research (AADR) and the International Association for Dental Research (IADR) from 1984 to 1996 (Fig. 1). Fig. 2 compares the percentages of the total number of presentations made by American

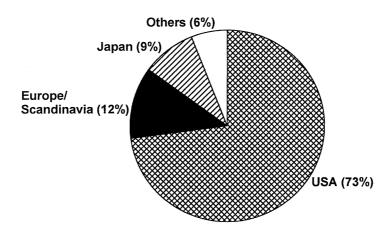
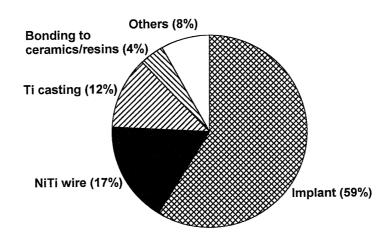


Fig. 2 Breakdown (percentages) of presentations on titanium at AADR and AADR/IADR meetings (1984-1996) by country.

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Fig. 3 Breakdown (percentages) of research areas for titanium presentations by American researchers (1984– 1996).

researchers and those from other countries for this period. These numbers were obtained from the presentations listed in the meeting abstracts for groups such as Dental Materials, Prosthodontics and Implantology. Since the AADR or AADR/IADR meeting is a major conference each year in these research areas, the trend seen in Fig. 1 can be considered a good indicator of the increase in research on titanium in the U.S.

Applications of titanium for dentistry have been slower to develop. However, during the last decade, the number of presentations related to titanium has steadily increased year by year at the AADR and AADR/IADR meetings (Fig. 1). Since the AADR is the largest division of the IADR and an AADR conference is held every year, it is not surprising that approximately three quarters of the total number of presentations related to titanium were made by American researchers (Fig. 2). Research presentations from Japan, whose research related to titanium casting has been very active, shared approximately 10% of the total number of presentations given since 1984. In Fig. 3, the trend showing the research areas of the investigations on titanium is given. Of the presentations made by American researchers, the majority (59%) have been related to implantology, followed by presentations on NiTi orthodontic wire (17%) and on titanium casting and properties of cast titanium (12%).

This review will provide information on the development of titanium in dentistry in the U.S., beginning with the first research done on oral titanium implants. The review will continue with a discussion of the progress made in titanium casting research on dental prostheses, orthodontic arch wires, soldering/welding and other related applications.

TITANIUM DENTAL IMPLANTS

Since the late 1980's, surface characterization studies of CP titanium and titanium alloys have been performed using state-of-the-art electron spectroscopic equipment because the surface

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condition of a titanium implant is a significant factor affecting the tissue reactions surrounding these implants. Two American research groups, Keller's group at the University of Iowa and Lucas' group at the University of Alabama at Birmingham have actively investigated the surfaces of titanium implants. Keller and coworkers characterized the CP titanium surface after applying various surface treatments including sterilization, a cleaning process and acid passivation^{6,7)}. The sterilization procedures examined did not reveal a noticeable variation in the surface morphologies and chemical composition of the surface film. The acid-passivated surface was covered with an approximately 3 nm-thick titanium oxide film on CP titanium⁸⁾ and an approximately 8 nm-thick film on the Ti-6Al-4V⁹⁾. Plasma cleaning (radio-frequency glow discharge) produced a slightly thicker oxide film (5nm) than other sterilization methods¹⁰⁾. A drastic decrease in the contact angle of deionized water (53 *vs.* 18°) was found on surfaces treated with this method. After several surface treatments, the surfaces of Ti-6Al-4V alloy were reported to follow trends similar to those for CP titanium¹¹⁾.

Lucas and co-workers¹²⁾ found that the anodization process increased the thickness of the oxide films formed as much as 4 times and improved the corrosion resistance; however, this surface film was found to be easily removed by the film bonding test. XPS experiments indicated the presence of adsorbed OH species and phosphorous in the surface film on the anodized specimen¹³⁾. The surfaces of CP titanium were examined after immersion in a physiologic solution (α -Minimal Essential Medium)¹⁴⁾. This study revealed that amorphous oxides and calcium phosphate compounds had formed on the titanium surface regardless of the amount of roughness present.

Cellular and tissue responses to titanium and titanium alloy implant materials have been extensively investigated by both *in vitro* and *in vivo* experiments. Cell attachment to surfaces has been investigated using fibroblasts, epithelial cells and osteoblast-like cells. The group at the University of Iowa has conducted a series of studies on cellular and tissue responses on the titanium surface along with their surface characterization. Cell type, sterilization and cleaning procedures, and surface topography were reported to affect cell attachment to the titanium surface and cell activity^{15,16}. Autoclaving the titanium implants decreased the number of fibroblast or osteoblast-like cells attached¹⁷⁻¹⁹. A sandblasted or rough surface revealed higher levels of cell attachment by osteoblast-like cells and gingival fibroblasts compared to a smooth surface²⁰⁻²³. On the other hand, epithelial cells behaved differently²²⁻²⁴. Recently an implant made from Ti-13Nb-13Zr was found to have a higher affinity for periodontal tissue cell attachment than a CP titanium or Ti-6Al-4V alloy implant²⁵.

The behavior of bacteria attached to the titanium surface was investigated by several different research groups under both *in vivo* and *in vitro* conditions. The *in vivo* investigations showed that specific *Streptococci* selectively adhered to the surface^{26–28)}. The biofilm formed on the titanium surface from contact with saliva or serum decreased bacterial attachment to the titanium surface^{29–31)}. Inconsistent results were obtained from *in vivo* studies which examined the effect of surface roughness on bacterial attachment; Patel *et al.*³²⁾ reported that the effect of surface topography on the bacterial attachment to the titanium surface. On the other hand, Wu-Yuan *et al.*³³⁾ found more

bacterial adhesion on sandblasted surfaces compared to smooth surfaces. Further, Yliheikkila *et al.*³⁰ indicated that the level of bacterial attachment to a rough surface was dependent upon the presence or absence of a saliva-coated layer.

Several factors affecting *in vitro* mineralization have been investigated; the autoclaving process³⁴, calcium ion implantation³⁵ and an excess number of titanium ions in an ambient environment³⁶ all had a deleterious effect on the mineralization *in vitro*. The surface topography is known to affect osteoblast-like cell activities; however, it is interesting that *in vitro* mineralization responses were found to be independent of the surface roughness³⁷. The research group at the University of Kentucky developed the electrodischarge compaction technique for fabrication of solid-core porous-surfaced implants using a Ti-6Al-4V alloy³⁸. This implant demonstrated rapid bone ingrowth and favorable osseointegration behavior^{39,40}. Later they fabricated a totally porous titanium alloy implant using this method. This totally porous implant revealed a significant increase in bone ingrowth with time⁴¹.

Coating hydroxyapatite or calcium phosphates on titanium or titanium alloy has become popular for implant materials⁴²⁾. Lucas and Lacefield and researchers at the University of Alabama at Birmingham have investigated the hydroxyapatite coating on titanium implant surfaces^{43,44)}. The shear strengths of the plasma-sprayed hydroxyapatite coating on titanium alloy were determined to be 15–17 MPa. The bond strengh decreased with increased immersion time because of degradation in the coating layers with time^{44,45)}. A research group at Ohio State University found a limited interdiffusion zone of titanium and calcium at the interface of the plasma-sprayed coating⁴⁶⁾. Other coating techniques, the ion-beam sputter deposition coating of calcium phosphates^{47,48)} and hydroxyapatite coating by electrodeposition⁴⁹⁾ have been studied.

At the 1996 AADR/IADR meeting, there was a presentation on a method of evaluating the fatigue resistance of CP titanium⁵⁰. This study was performed because concern has increased about the fatigue failure of implants. The application of marker blocks during fatigue testing was reported to provide useful information about implant fatigue crack initiation.

DENTAL TITANIUM CASTING

Another area of dental titanium study attracting interest in the U.S. is research related to casting technology. In 1959, Beder and Ploger⁴⁾ suggested using a centrifugal casting machine with induction heating housed in a vacuum chamber to cast titanium implants. However, they never pursued their research by actually constructing the equipment. It was not until around 1977 that experimental castings of titanium for crowns and partial denture frames were finally made by induction melting in vacuum as Beder and Ploger had earlier suggested⁵¹⁾. Waterstrat and his associates^{51,52)} at the National Bureau of Standards consulted Schuyler and others at the Garrett Corporation in Arizona about casting dental prostheses with a titanium alloy containing 13 wt% Cu and 4.5 wt% Ni. Schuyler *et al.*⁵³⁾ had already performed extensive studies on the casting of industrial titanium alloys. This alloy was chosen since its fusion temperature is much lower than that of CP titanium (1329 *vs.* 1670°C);

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it was hoped that oxidation during melting and casting and the reaction of the molten metal to the investment material would be minimal. Waterstrat continued his pioneering research efforts on several projects, but his experiments gradually tapered off, mainly because of lack of support from funding agencies. Notable among his work was the construction of specially designed, split-chamber argon-vacuum arc casting equipment which was patented in the U.S. in 1985⁵⁴). His idea of using this equipment for titanium casting with magnesia investment was presented at the IADR/AADR meeting in 1985⁵⁵). He and his associates⁵⁶) also presented a clinical trial of titanium crowns placed in patients for as long as 22 months and reported that, although there were problems in the fit of the crowns and some wear on the opposing tooth, patients did not experience any unusual taste or discomfort from the titanium crowns compared to gold crowns. Later Waterstrat was involved in the examination of the properties of commercially available investment materials for titanium with Mueller *et al.*^{57,58)} at the American Dental Association in Chicago. They reported complex reactions by the investment material (Biovest, Dentsply) which contained cristobalite, but less reaction from a silica-containing investment (Rema Exact, Dentaurum).

Other attempts to research titanium for dental casting began in 1984 by Greener and colleagues at Northwestern University. Their research was initiated at first to develop titanium alloys with a low fusion temperature and focused on finding an alloy with corrosion resistance and biocompatibility equivalent to CP titanium. Alloys they examined included various binary alloys such as Ti-Cu, Ti-Co, and Ti-Ni⁵⁹⁾, Ti-Pd⁶⁰⁾ and ternary titanium alloys including Ag and Cu⁶¹. While they found that the elctrochemical behavior of the alloys was comparable to pure titanium, they reported much increased hardness of most of the alloys examined. In 1985, Greener's group began to examine the capabilities of two commercial titanium casting machines acquired from Japan, the Titaniumer made by Ohara and the Castmatic, by Iwatani. In order to minimize the reactions between the investment materials and titanium, they first utilized the "face-coating" technique prior to investing in a phosphate-bonded silica material using yttrium oxide, zirconia or titanium dioxide with a zirconium acetate binder⁶²). Using the Castmatic, they cast alloys which contained V (15%), Cu (20%) or Pd (30%), including Ti-6Al-4V alloy and pure titanium. They noted a considerable increase in the strength of the castings, which consisted of quasi-equilibrium phases created by alloying. In corrosion tests, a strong passive trend was seen. Their group also examined the accuracy of fit of pure titanium on MOD inlay dies using five different investment materials⁶³⁾. According to Greener⁶⁴⁾, quantitative and statistical treatments found that all castings were undersized by at least 100 μ m. Later, they tried incorporating Zr powder into the investment material in order to increase expansion, a method that had also been tried in Japan.

The research team at Northwestern is still continuing their initial efforts to study titanium castings from various aspects. These include comparative studies of strength properties⁶⁵, castability⁶⁶, and electrochemical behavior⁶⁷ of pure titanium and Ti-6Al-4V alloy. They also initiated an examination of bonding experimental porcelains to cast titanium or titanium alloys⁶⁸⁻⁷¹. They claimed that experimental SiO₂/Al₂O₃ ceramics bonded well to the castings. In 1991, Tamaki *et al.*⁷² examined the effect of two commercial investment materials on the casting accuracy of pure titanium using different types of

crucibles. They reported that the types of investment materials significantly affected the fit of MOD inlays but the types of crucibles used did not. In another more recent study from the same group, Pang *et al.*⁷³⁾ baked Porcera porcelain on milled titanium and found acceptable bonding, whereas Gilbert *et al.*⁷⁴⁾ reported that effective bonding strengths for the porcelain baked could be obtained using a titanium bonding agent.

More recently, a research group at the University of Texas Health Science Center at San Antonio became involved in research on dental titanium. In 1989, Blackman *et al.*⁷⁵⁾ presented a 90% success rate for a cast titanium RPD pattern based on the observation of voids, peripheral irregularities, etc. Other studies of pure titanium castings at San Antonio include evaluations of marginal fit⁷⁶⁾, casting accuracy⁷⁷⁾, the effect of investment materials on strength⁷⁸⁾, and castability⁷⁹⁾ and sprue design^{80,81)}. One of the interesting studies being done is the x-ray radiographic examination of internal voids of titanium castings; these studies reported on the appropriate radiographic operating conditions for examining castings of various thicknesses⁸²⁾. Baez's examination⁸³⁾ of various commercial ceramics for cast titanium reported promising results of this technique.

There is notable work being performed at other research sites in the U.S. One of the American dental companies, Jelenko, developed titanium casting equipment in early 1990 called Ticast 3000, which uses a helium arc-melting/centrifugal rotating system^{84,85)}. Other research and experiments in the U.S. have been carried out by Mueller and Fan⁸⁶, who examined the permeability of various investment materials, including two commercial materials for titanium casting by the American Foundrymen Society's standard air permeability. They found that, even with the very low standard permeabilities of dental investments, significant differences in the permeability levels exist among the investment materials. Chai and Stein⁸⁷⁾ in Boston reported consistent castings of multiple three-unit crowns in the Cyclarc machine (Morita) with Titavest investment. Earlier, this group examined the marginal fit of pure titanium casting crowns⁸⁸⁾. Liles et al.⁸⁹⁾ compared the electrode potentials of seven different dental alloys with pure titanium and found that except for a Ni -Cr-Be alloy, all other alloys are cathodic to titanium. Oshida and Hashem⁹⁰⁾ found that nitriding titanium was effective for producing stronger bonding to porcelain. More recently, Wang and Welsch⁹¹⁾ reported that in their preliminary study on producing a better oxide layer for the porcelain veneering on titanium, the oxide adherence of titanium coated with Pd or Cr was superior to the uncoated titanium.

Among the 1996 AADR/IADR presentations, there are several reports on titanium casting. The accuracy/fit of cast CP titanium crowns was examined by Blackman's group at the University of Texas Health Science Center at San Antonio⁹²⁾. When pressure difference casting equipment was used, there was still room for improvement in obtaining acceptable crown margins. A comparison of castability using the mesh pattern method in two different types of equipment (single-chambered and double-chambered pressure difference machines) reported a 99.30% castability rate for the single-chambered *vs.* 48.56% for the double-chambered equipment⁹³⁾. In a collaborative study at the University of Texas Health Science Center at San Antonio and the Lackland Air Force Base Medical Center, Sutton *et al.*⁹⁴⁾ compared the porosity of CP titanium RPD frameworks made with five different sprue designs using the digital image analysis of radiographs.

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the difference in sprue design did not significantly affect the porosity values, it did affect the castability of the fine peripheral areas. In addition, Baez *et al.*⁹⁵⁾ at San Antonio also reported on the effect of the sprue design on the mesh pattern filling using two different pressure difference machines and indicated the importance of the sprue design for the casting equipment they employed.

The evaluation of titanium alloys for dental casting was performed by a group at Case Western Reserve University⁹⁶⁾. Examinations of Ti-Ag and Ti-Ag-Cu alloys with some heat treatment procedures showed that the alloys were strong enough but needed more ductility. At Baylor College of Dentistry, the Texas A&M University System, a research team recently initiated titanium casting research. Watanabe *et al.*⁹⁷⁾ reported on initial work using a NIOM-Baylor experimental pressure difference casting unit to find the optimal operating conditions for producing sound CP titanium castings. This study was the continuation of collaborative work on titanium casting with NIOM and Kyoto University.

For the corrosion behavior examination of pure titanium, Gilbert and Mante⁹⁸⁾ conducted a comparative electrochemical study on high-purity polycrystalline and single-crystal titanium. The single-crystal titanium prepared by Mante *et al.*⁹⁹⁾ showed one order of magnitude better corrosion current in the single-crystal titanium with some crystallographic orientation dependence.

A group at the University of Michigan has been examining PMMA-wrought titanium rod bonding. The most recent study by May *et al.*¹⁰⁰⁾ revealed increased bonding when using surface-treated titanium (treated with alumina air abrasion plus silane and an opacifier). Their study was the extension of earlier work by May *et al.*¹⁰¹⁾. This same group also reported in 1993 that titanium could be effectively bonded with All-Bond 2 and Panavia and suggested the potential use of these adhesive cements for bonding Maryland bridges¹⁰²⁾.

ORTHODONTIC ARCH WIRES

Since the introduction of shape-memory or superelastic NiTi arch wires to orthodontics in 1971, NiTi arch wires have been investigated in order to characterize their mechanical properties, phase transformation and structures. In the U.S., two types of commercial titanium alloy wires are currently available : beta-titanium alloy (Ti-Mo based alloy) and NiTi alloys. A large number of titanium alloy wires are currently on the U.S. market. Burstone and Goldberg at the University of Connecticut investigated the properties of beta -titanium alloy wires for orthodontic purposes in the late 1970's and 1980's¹⁰³⁻¹⁰⁵. The majority of recent studies have concentrated on the commercially available NiTi wires. The phase transformation behavior of commercial arch wires has been examined by Differential Scanning Calorimetry or x-ray diffractometry¹⁰⁶⁻¹¹². Sachdeva and co-workers¹¹³⁻¹¹⁵ at Baylor College of Dentistry characterized various aspects of the mechanical and corrosion behavior of NiTi alloy wires in collaboration with Indiana University and the University of Tsukuba, Japan. The reported studies suggested that there is a large variation in the properties of the NiTi arch wires on the current market¹¹⁶⁻¹¹⁸. Recently, a potential of Ti -Nb archwire was suggested for use as a finishing wire in orthodontic treatment¹¹⁹.

SOLDERING/WELDING

Finally, a brief word about soldering, which is often used in prosthodontic dentistry for constructing and joining dental appliances. This procedure is very important for titanium castings. Thus far, some research groups have investigated the properties of soldered¹²⁰⁻¹²² or welded¹²³⁻¹²⁵ titanium metals. Since 1992, the research team¹²⁶ at the University of Washington has observed the clinical performance of laser-welded implant frameworks which were developed in Sweden (Porcera system, Nobelpharma). Their one-year report revealed favorable results, and they continue to follow up the clinical performance of this technology. Welding or soldering is an area of dental titanium technology which has not yet been explored in depth.

SUMMARY

In summary, we believe the future of dental titanium technology looks bright. The potential for wide-spread use of titanium in dentistry is there; however, very little systematic research on dental applications of titanium has been made in the U.S. More extensive research on implantology, casting technology, and other applications must be done in the U. S. and elsewhere in the world before this interesting metal can be fully utilized. Japan is currently the most advanced and active country in the research and application of titanium in dentistry. Other dental materials researchers and clinicians in the world encourage Japanese researchers to present their studies more often at international meetings in order to share with the world their top-notch knowledge in the application of titanium.

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本号掲載論文の和文抄録

歯科用チタン:アメリカにおける研究の推移

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近年, チタンは材料学研究者や臨床医に大きく注目を 浴びてきている.アメリカでのチタンの生体材料として の歴史は,1940年にチタンインプラントの動物実験が行 われた時から始まる.その後,1959年,口腔内インプラ ントを作製するにあたりチタン鋳造の必要性をアメリカ の Bedger と Ploger がはじめて唱えた.1977年には最 初のチタン鋳造による歯科補綴物が作製する試みがなさ れたと報告されている.過去10年間, IADR および AADR においてチタンに関連する発表数は毎年増加し 続けている。大多数の研究発表は、アメリカからの研究 であるが、そのうち半数以上は歯科用インプラントに関 連したものである。チタン鋳造や補綴物としての応用に 関するアメリカからの報告は、近年増加しているものの、 今のところ数が限られている。このレポートは、アメリ カにおける歯科用チタンの発展と研究の動向を報告す る。

磁石と吸引する試作コンポジットレジンの性質

平野 進,安川宏美,野本理恵,森山圭介,平澤 忠 鶴見大学歯学部歯科理工学教室

SUS 444 ステンレス鋼フィラーを含み、磁石と吸着す る化学重合型コンポジットレジンを作製した.フィラー には種々の濃度のシラン処理を施した.このコンポジッ トレジンは操作性がよく,保存安定性に優れていた.もっ とも良い性質を示したコンポジットレジンは以下のよう であった.市販磁性アタッチメント用の磁石に対する吸 引力は磁石附属のキーパシステムのそれの1/3-1/4 で

あった。曲げ強さとヌープ硬さは各々76 MPa (7.7 kgf/ mm²)ならびに 64 KHN で,市販の化学重合型コア用コ ンポジットレジンよりも低い値であった。コンポジット レジンからの溶出金属量は 1 %乳酸溶液 7 日間浸漬の結 果 0.7 mg/cm² で,0.9%NaCl 溶液 7 日間浸漬では検出 できなかった。

フィラー形状および粒径が試作光重合型コンポジットレジンの 機械的性質に及ぼす影響

宮坂 平

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平均粒径 1.7~21.5 μm の無定形フィラー 4 種類, 平 均粒径 0.46~31.2 μm の球形フィラー 5 種類および平 均 0.04 μm のミクロフィラーのうちから, それぞれ 2 種 類づつを組み合わせて混合した二成分系のハイブリッド 型フィラーを用い光重合型コンポジットレジンを試作 し,圧縮強さおよび間接引張強さを測定した.この結果, 無定形フィラーと球形フィラーを混合すると,組み合わ せるフィラーの粒径が小さいほど強度は大きくなる傾向