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Some surface characteristics of composite resin filling materials*

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Keywords: Composite resins; filling materials, materials, resins

SUMMARY

Twelve composite resin filling materials were subjected to hardness tests and their surfaces were examined by scanning electron microscopy. An ultraviolet source used to polymerize one of the materials was examined. The hardness of the materials varied considerably . Spherical filler particles were not as well retained as irregularly shaped filler particles, but retained filler particles project from surfaces. Capsulated resins had fewer air inclusions than hand-mixed resins. The ultraviolet source had similar characteristics to earlier devices. A newly-available resin with no inorganic filler was examined. It has a relatively low hardness and a very smooth surface.

OPSOMMING

Die hardheid van twaalf saamgestelde herstellingsharse is getoets en hul oppervlaktes met 'n aftaselektronmikroskoop ondersoek. Die ultraviolet ligbron waarmee een van die materiale gepolimeriseer is, is ook ondersoek. Die hardheid van die materiale het aansienlik verskil. Balvormige vullerdeeltjies is swakker geheg as onreelmatige deeltjies, maar behoue vullerdeeltjies steek aan die oppervlaktes van die materiale uit. Minder lug is ingesluit in die harse wat in kapsules voorsien word en meganies gemeng is, as in materiale wat met die hand gemeng is. Die ultravioletbron toon soortgelyke kenmerke as vroeere toestelle. 'n Pas beskikbaargestelde hars, sonder enige anorganiese vulmateriaal, is ondersoek. Dit is betreklik sag en toon 'n besonder gladde oppervlakte.

INTRODUCTION

Composite resin filling materials have attained much popularity in the last decade. They have, however, been a source of disappointment in some respects and, in particular, the surfaces often show wear and roughness. Rough surfaces are undesirable on dental restorations because roughness can cause:

(1) mechanical irritation of soft oral tissues;
(2) cosmetically unacceptable staining;
(3) plaque and food debris retention; or
(4) tarnish and corrosion (in metal restorations).

The fact that these resins exhibit excessive wear when exposed to masticatory function has been observed in clinical studies by Phillips *et al* (1973) and by Osborne, Gale and Ferguson (1973). Even on surfaces where the material is not subject to direct masticatory force it loses its harmonious appearance and tends to stain. Staining is generally accepted as resulting from roughness of the surface of composite resins, which is caused by four factors, namely:

(1) voids resulting from inclusion of air during mixing; contact with the matrix and adhesion thereto;
(2) voids resulting from inclusion of air during mixing;
(3) voids resulting from loss of particles of inorganic filler; or
(4) projection of filler particles.

Larato (1972) found that gingival tissue related to composite resin restorations developed gingivitis and the restorations retained plaque.

It has been stated that the best surface finish on a composite resin is attained by permitting the resin to set in contact with a matrix and the procedure has been advocated by manufacturers. That view has also been supported by writers such as Chandler, Bowen and Paffenbarger (1971), Messing and Ray (1972) and Deubert and Jenkins (1972). Such a surface may, nevertheless, be far from ideally smooth. For instance, Hannah and Smith (1973) found surfaces damaged by adhesion to matrix material and by voids. Voids up to 10μm in diameter were found by Gray and Gavin (1975). Johnson, Jordan and Lynn (1971) observed irregular matrix-formed surfaces which they attributed to polymerization shrinkage.

* Based on papers read at meetings of the South African Division of the International Association for Dental Research, Durban, 1976 and Stellenbosch, 1977.

It is widely believed that there may be a relationship between hardness and abrasion resistance. It is difficult to demonstrate such a relationship and sometimes possible to demonstrate that softer materials possess superior wear resistance. For example, Shell and Hollenback (1966) demonstrated that the rate of wear of hard gold alloys is greater than that of softer alloys. Filled resins are harder than unfilled resins (von Fraunhofer, 1971). Lugassy and Greener (1972) found that the abrasion rate of filled resins was greater than that of unfilled resins and suggested reasons, including the possibility that displaced filler particles themselves act as an abrasive. The relationships between *in vitro* and *in vivo* studies of wear are not always clear (Phillips, 1975; Craig and Powers, 1976).

It appears that the abrasion resistance of a composite resin may be high if the water sorption of the material is low (British Patent, 1972). The filled Bowen type resins absorb much less water than polymethal methacrylate and less than a filled methacrylate (Braden, Causton and Clarke, 1976). Harrison and Draughn (1976) found no consistent relationships between abrasive wear, tensile strength and hardness of composite resins. Heath and Wilson (1977) consider that water absorption affects the wear of composite resins by loosening the bond between the filler particles and the resin matrix.

Clinicians recognize that composite restorations usually have to be trimmed. Hence manufacturers have developed a variety of trimming and finishing devices for these materials and the effects of different finishing techniques have been studied by scanning electron microscopy (Johnson *et al*, 1971; Chandler *et al*, 1971; Gray and Gavin, 1975; McLundie and Murray, 1972; Hannah and Smith, 1973). Finish of composite resin has also been studied with a surface roughness measuring machine (Glantz and Larsson, 1972). As trimming is needed in clinical practice it was decided to examine the hardness and the microscopic appearances of some trimmed samples of composite resins, all finished by a uniform technique.

MATERIALS AND METHODS

Twelve composite resin mterials were examined. They are identified in Table I. All have inorganic fillers and most are of the Bowen type (bis-GMA), but material 1 is a polymethyl methacrylate with a tri-n-butylborane catalyst and material 2 is a methyl methacrylate with methacrylic acid. Material 5 is based on a urethane dimethacrylate system and material 7 has its shade determined by the selection of different liquid catalysts. Material 11 is ultraviolet-polymerized and is a single paste which does not require the addition of a separate catalyst, unlike earlier ultraviolet-polymerized resins, and is supplied with an unfilled ultraviolet-polymerized resin which serves as a primer, a glaze, or a fissure sealant. Material 12 is a diacrylic ester of a bifunctional alcohol (British Patent, 1972) supplied in a novel syringe-capsule (U.S. Patent, 1975).

Samples of each material 1 cm x 1 cm x 0,5 cm were prepared, using simple L-shaped metal moulds, in which the materials were formed against Du Pont MYLAR* sheet, supported by a glass slab. The materials were mixed according to the makers' instructions. SEVRITON FILM** was used as a separating agent to prevent adhesion to the moulds. The specimens were stored in deionised water at 37°C until required for use.

Five samples of each resin were prepared for hardness testing. The smooth surface formed against MYLAR sheet was reduced by grinding with a grain 400 silicon carbide abrasive disc, on a DAP-2 polishing machine***. The disc was operated in one direction relative to the specimen, to "grain" the surface, facilitating subsequent observation. The specimens were each subjected to a load of 1 kN for 15 sec, using a 5 mm diameter Brinell ball, in a Type W ten-

* Marshall Hinds (Pty) Ltd., Johannesburg, S.A.
** Amalgamated Dental, London, England.
*** Struers Scientific Instruments, Copenhagen, Denmark.

Table I. Identification of materials

No.	Brand.	Batch No.	Maker.	Type.
1	POLYCAP	020771/ 070771	VIVADENT, Schaan, Liechtenstein.	Capsulated
2	TD 71	003CD 002AD (liquid)	Dental Fillings, London, England.	Capsulated
3	CONCISE	2291-02(K)	3M Co., St. Paul, U.S.A.	Two pastes
4	ADAPTIC	2L 037	Johnson & Johnson, New Brunswick, U.S.A.	Two pastes
5	COSMIC	PE 34 (base) PE 33 (catalyst)	Amalgamated Dental, London, England.	Two pastes
6	ESTIC	51206 (base) 51113 (catalyst)	Kulzer, Bad Homburg, W. Germany.	Two pastes (tube catalyst)
7	DRS	60593 (base) 60491 (catalyst)	Oratec, Santa Ana, U.S.A.	Paste with liquid catalyst
8	COMPOCAP	040774	VIVADENT, Schaan, Liechtenstein.	Capsulated
9	COMPOCAP-S	171176 355	VIVADENT, Schaan, Liechtenstein.	Syringe-capsulated
10	EPOLITE 100	JM 16	G-C Dental Industrial Corporation, Tokyo, Japan.	Two pastes in syringes.
11	ESTILUX	601025 610024	Kulzer, Bad Homburg, W. Germany.	One paste UV in syringe
12	NIMETICAP	C 139/26	ESPE Gmbh, Seefeld/Oberbay, W. Germany.	Syringe-capsulated

someter****. The diameters of the indentations were measured immediately after the removal of the load, in order to minimize the possibility of inaccuracies resulting from elastic recovery. The Brinell Hardness Number (BHN) was calculated and averaged for the five specimens of each material.,

Specimens of each material were examined by scanning electron microscopy. Since it is usually necessary in clinical practice to trim and finish resin restorations the MYLAR-formed surface of each specimen was reduced on the DAP-2 polishing machine, using a grain 800 silicon carbide abrasive disc, after which the ground surface was polished with a bristle brush and a slurry of fine pumice and water and finished with a soft horsehair brush and a slurry of whiting and water. After drying the specimens were coated with carbon (at 100 millitorr) and gold/palladium (at 75 millitorr) in a Technics Hummer II sputter coating unit*. The prepared specimens were examined in a Cambridge Stereoscan S4 scanning electron microscope** operated at 20 kV with a tilt angle of 45°.

The DURALUX UV-20 ultraviolet source*** used to polymerize material 11 was examined using a spectroscope**** and fluorescent screens.

Subsequent to the completion of the investigation of the composite resins listed in Table I a new material, ISOSITE*****, became available. This material is stated by the makers to contain an organic filler and no inorganic filler. It was subjected to the same examination as the other materials. ISOSITE was used in the form of a two paste system, base and catalyst, supplied in syringes and named ISOPAST.

The ISOPAST used was

Base (Shade 35) Batch No. 040 477
Catalyst Batch No. 280 377

**** Mo[illegible]

* Technics, Alexa[illegible]
** Cam[illegible]dge Scient[illegible] Instruments Ltd., Cambr[illegible] England.
*** Kulzer, Bad H[illegible] W. G[illegible]
**** Hilger, Margate, E[illegible]land
***** VIVADENT, Schaan, Liechtenstein

RESULTS

The results of the hardness tests are shown in Table II. The low BHN for material 6 is not considered valid, as this material did not appear to polymerize satisfacto in the bulk used to make samples, althou polymerized satisfactorily in clinical quantities. One specimen of material 4 gave a BHN of 89, the only result not close to the average for that material. Close examination of the specimen disclosed that the indentation had been made on an area where a whitish streak appeared (Fig. 1). The result was discarded, the test repeated on a fresh specimen, and the new result, being close to the others, was accepted. Examination of MYLAR-formed surfaces revealed whitish streaks in specimens of materials 3,4,5,7 and 10.

Scanning electron microscopy showed numerous voids in material 1 (Fig. 2). These voids appear to be the result of loss of spherical filler particles, rather than to air inclusions, since the voids are of comparabe size to the filler particles. Other materials, with irregular filler particles, showed infrequent loss of filler particles, (Fig. 3), although the filler particles projected from the surfaces (Fig. 4). Materials 3,4,5,7 and 10 showed numerous voids apparently resulting from air inclusions (Figs. 5 & 6). Material 11 showed three rather large defects (Fig. 7), probably the result of poor adaptation to the MYLAR sheet. Materials 1,2,6, and 11 showed few voids from air inclusions, which were mostly very small (Fig. 8). Materials 8 and 12 (Fig. 9) were almost completely free of voids, whilst material 9 showed a moderate number of very small voids (Fig. 10).

The ultraviolet light source emitted strongly through

Table II Brinell Hardness [illegible] materials

Material	BHN
1. POLYCAP	19
2. [illegible]	29
3. CONCISE	48
4. ADAPTIC	[illegible]
COSMIC	55
6. ESTIC	32
DRS	45
8. COMPOCAP	64
9. COMPOCAP-S	76
10. EPOLITE 100	65
11. ESTILUX	63
12. NIMETICAP	72

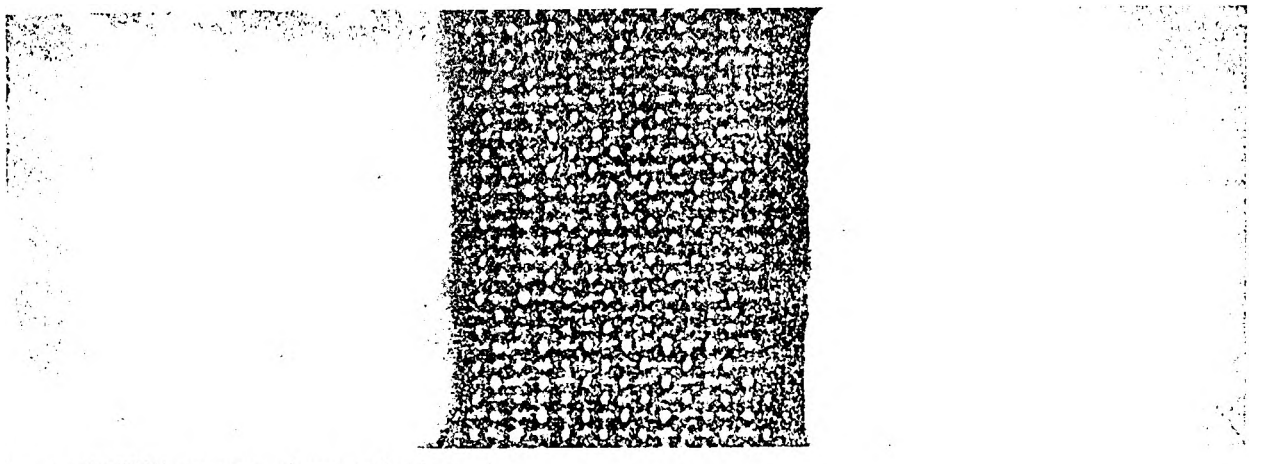

Fig. 1 ADAPTIC specimens showing whitish streaks.



Fig. 2. POLYCAP SEM x 100



Fig. 4. NIMETICAP SEM x 1000



3. NIMETICAP SEM x 200

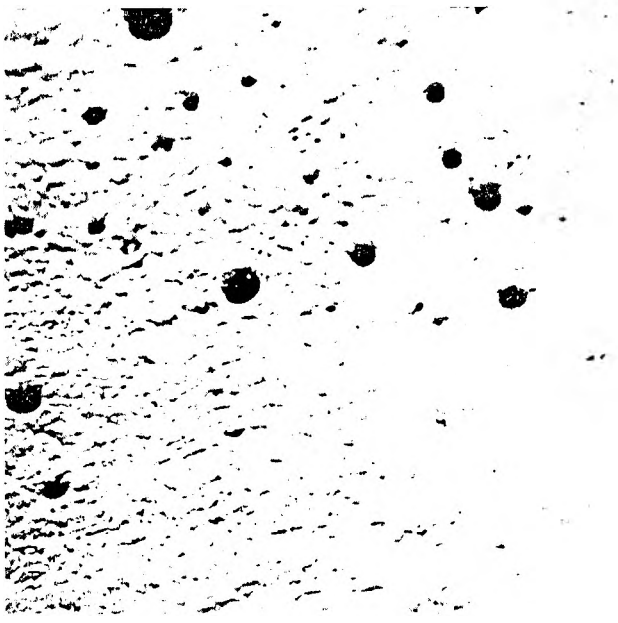

Fig. 5. ADAPTIC SEM x 50

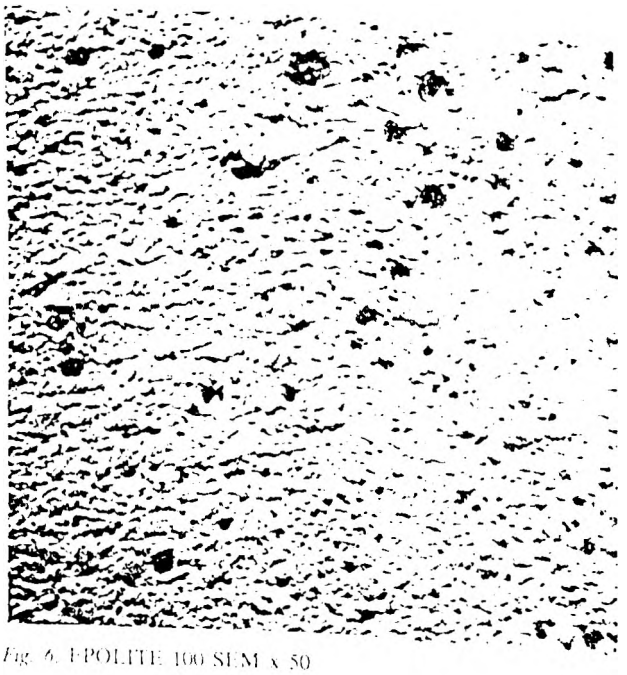

Fig. 6. I-POLITE 100 SEM x 50

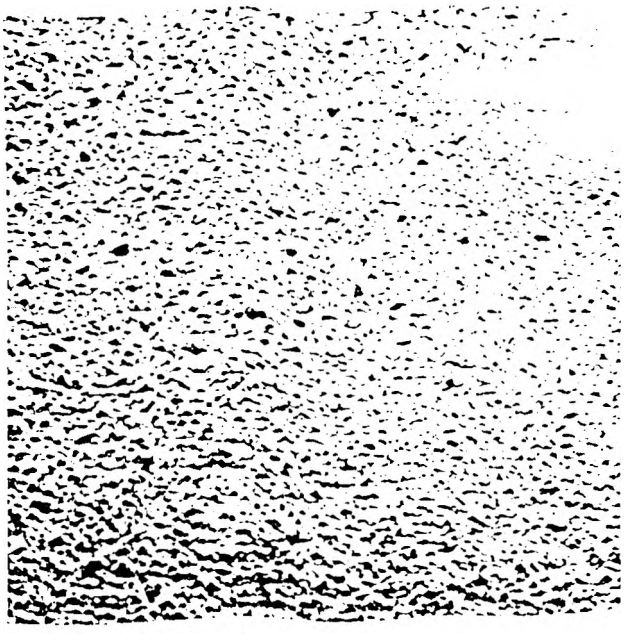

Fig. 8. I-STIC SEM x 50

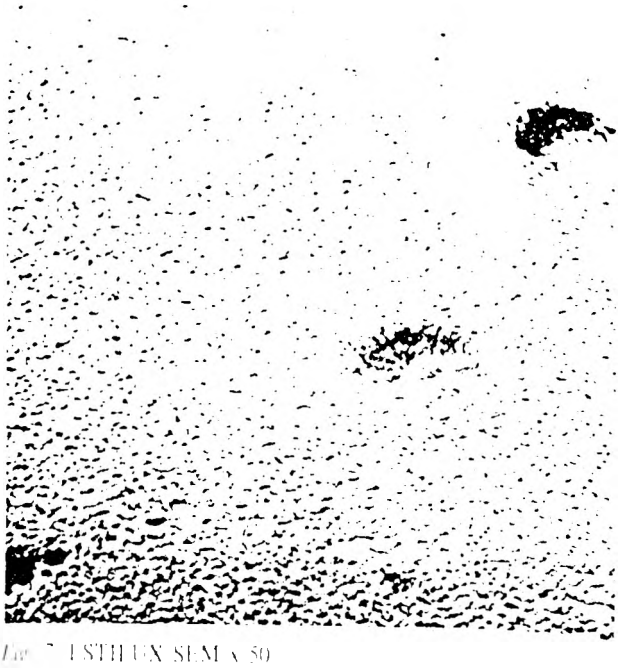

Fig. 7. I-STELUX SEM x 50

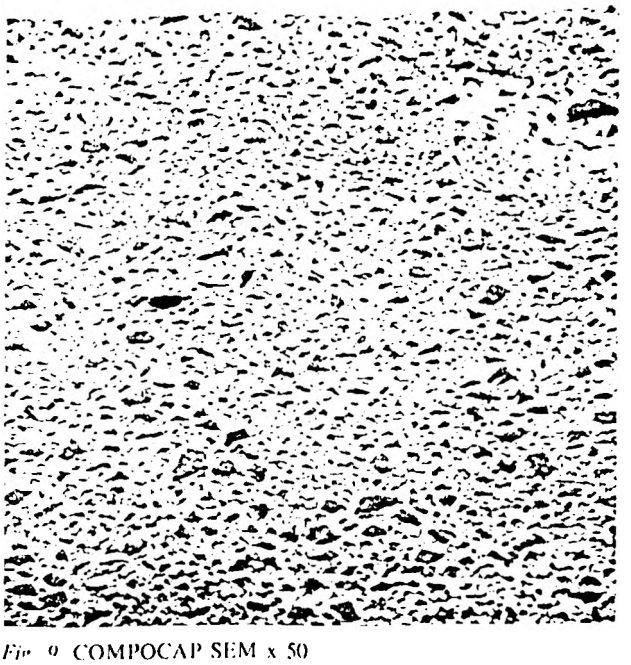

Fig. 9. COMPOCAP SEM x 50

filtered exit window of the light guide between 362 and 365 nm and also between 400 and 440 nm and there was slight emission at 333 nm (Fig. 11). Slight leakage of visible light was noted from two ventilation grids at the back of the lamp housing and from the cover of the lamp. This did not cause fluorescence when examined in total darkness with fluorescent screens.

ISOPAST had a BHN of 35 and at low magnification (Fig. 12) a number of small voids were evident, while at higher magnification no filler particles were to be seen, although polishing scratch marks were conspicuous (Fig. 13).

DISCUSSION

The results of the hardness tests show that materials 1 ' 2 are not very hard, compared to the other materials, whilst materials 9 and 12 are somewhat harder than the majority of the materials. As has been noted, the clinical significance of hardness is uncertain.

When two-paste composite resins packed in jars were first introduced the manufacturers advocated thorough stirring of the pastes before use. This is no longer the case. The whitish streaks seen in samples of five materials could be the result of settling of the fillers, or to uneven distribution of fillers for other reasons, or to incomplete mixing of base and catalyst pastes. The fact that whitish streaks were present in specimens of material 7 (Fig. 14), which is supplied with a liquid catalyst, suggests that incomplete mixing of pastes was not the cause of the whitish streaks. Whitish streaks were not observed in specimens prepared from any of the five capsulated resins examined. Whitish streaks are not observable in clinical quantities of resins

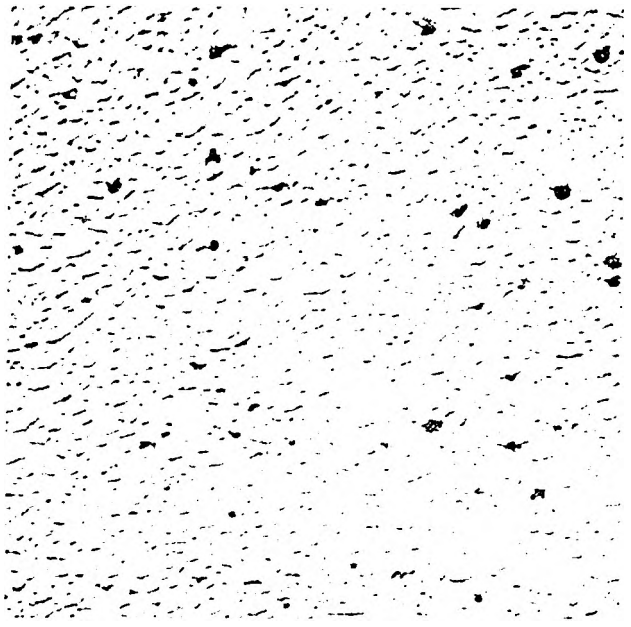

Fig. 10. COMPOCAP-S SEM x 50

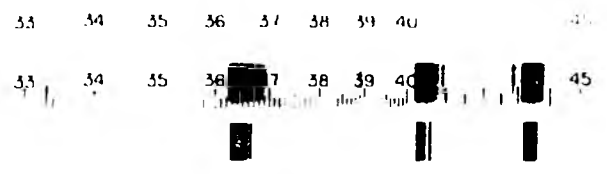

Fig. 11. Emission spectrum of DURAFIX UV-20

The examination of specimens by scanning electron microscopy shows that irregularly shaped filler particles are retained better than spherical particles, although the particles retained project and thereby contribute to the roughness of the surface. The presence of voids resulting from air inclusions was much more evident, both in regard to numbers and size, in the specimens of hand-mixed resins than in the specimens of capsulated resins. This observation corresponds with that of Beasley, Makinson and Rohr (1976), who found that "the maximum number of bubbles per unit volume for Compocap mixes was almost half the minimum for two non-capsulated composite resins.

In view of the foregoing it is considered that capsulated composite resins are generally superior to hand-mixed resins in at least two respects – they have fewer air inclusions, which in turn means a less rough surface, and they are also more homogeneous (lack of whitish streaks).

So long as inorganic fillers of the types currently used are present the surfaces of composite resins will be rough, either because of loss or projection of particles. Clinical experience shows that glazes are not durable and they can be damaged by tooth brushing (Makinson and Rohr, 1976). Another approach to the difficulty is the search for a better method of filler retention than silanization, which breaks down when water is present (Heath and Wilson, 1977). Bowen and Reed (1976, a and b) propose the use of semiporous filler particles.

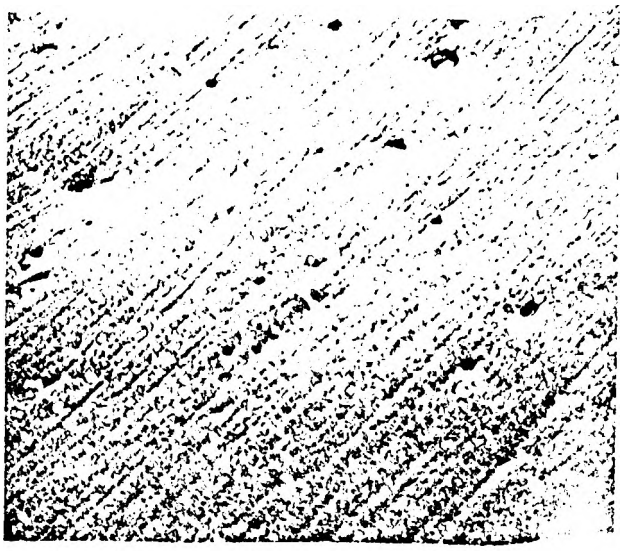

Fig. 12. ISOPAST SEM x 50

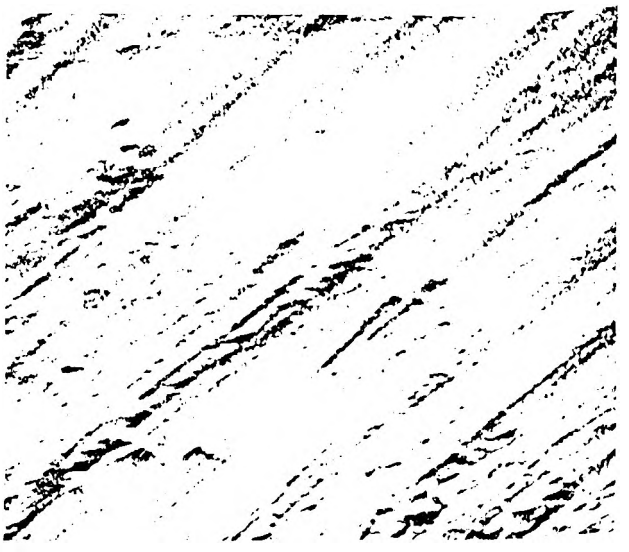

Fig. 13. ISOPAST SEM x 1000

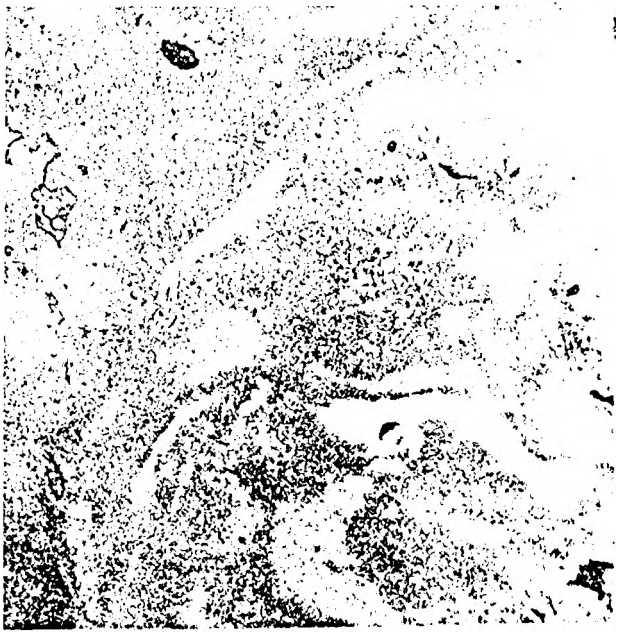

Fig. 14. DRS specimen showing whitish streaks.

Elimination of filler loss would still leave filler particle projection as a cause of roughness. A promising development is the marketing of the material with an organic filler which, in clinical trials, has maintained its contour and surface appearance better than materials with inorganic fillers (Mannerberg, 1977). The appearance of air bubbles in this material (Fig. 12) was to be expected in a handmixed resin and an improvement can be anticipated when it is available in capsules.

The emission spectrum of the DURALUX UV-20 ultraviolet source was comparable to those of earlier devices reported by Rock (1974) and the small amount of visible light which escapes is not considered harmful. All the same, users of ultraviolet devices should bear in mind the safety recommendations of Birdsell, Bannon and Webb (1977) and see that such devices are maintained in an efficient condition (Young *et al*, 1977).

ACKNOWLEDGEMENTS

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