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STRUCTURE STUDIES OF AMALGAM
I. CORRELATION BETWEEN STRUCTURE
AND PHYSICAL PROPERTIES

by

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In studying the mechanical and physicochemical properties of amalgam fillings it is often useful to know these properties in localized, small areas, such as the filling margins.

Swartz & Phillips (1958) carried out such studies by a chemical method of analysis described by *Crawford & Larson* (1955). A chemical approach is based upon the correlation between the chemical and physical properties of amalgam. Such a method, however, presents limitations for analysis of small samples. Besides, it is complicated to use in certain, more detailed studies.

Other authors (e.g. *S. Obst*, 1953) have used micromechanical testing methods for characterization of the chemical and physical properties of amalgam. However, according to experiments by one of the present authors (K.D.J.) these methods can scarcely give a detailed graduation of an amalgam filling, since quite considerable differences in physical properties were reflected as very moderate, often doubtful differences in the measuring results.

The aim of the present work is to present a method that, based on a microscopic examination of the structure, permits the greatest possible detailed and accurate characterization of the

mechanical and physicochemical properties of the amalgam to the extent they are correlated with the structure.

MATERIALS AND METHODS

The experiments were carried out with the following silver amalgam alloys: DAB Standard Alloy (A.B. Svenska Dental Instrument), Solila Alloy (Amalgamated Dental Trade Distributors, Ltd.), and True Dentalloy (S. S. White Dental Mfg. Co, G.B.). All three products meet the requirements of the American Dental Association Specification No. 1 for amalgam alloys. With regard to particle size they must be described as medium-grained. Only one batch number was used of each brand.

Table I

Average mercury content (%) of the amalgams as affected by the condensation pressure

Condensation pressure	10 kg	30 kg	60 kg	120 kg
True Dentalloy	54.0	49.4	45.2	39.2
Solila Alloy	51.6	47.2	42.4	36.8

Table II

Crushing strength (kg/cm²) of the amalgams as affected by the condensation pressure

Condensation pressure	10 kg	30 kg	60 kg	120 kg
True Dentalloy	3370±61	3622±119	3785±93	4136±87
Solila Alloy	2821±47	2901±98	3099±74	3442±119

From these alloys cylindrical specimens were prepared in a steel mold by a method previously described by *Jørgensen et al.* (1964). The specimens had a diameter of 5 mm and a length of 10 ± 0.5 mm. The condensing pressures were 10, 30, 60, or 120 kg. Trituration was done with a Wig-L-Bug mechanical amalgamator in Bakelite capsule with piston, and the normal mixing time was 15 seconds. Proportioning was in accordance with the

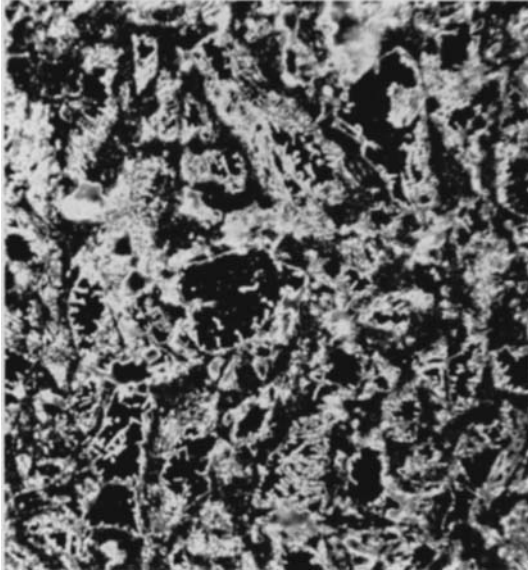


Fig. 1 A.

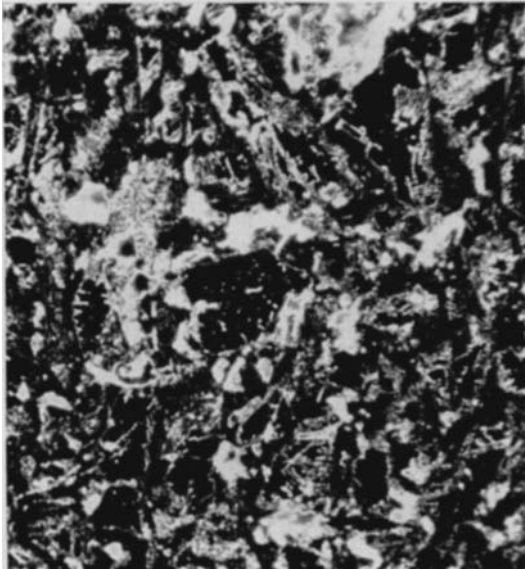


Fig. 1 B.

Fig. 1. The same amalgam specimen etched for 5 (A) and for 20 seconds (B). The two surfaces are identical in appearance. Note the black well-defined residual grains of the γ -phase. True Dentalloy, mercury content approx. 45 %. Magnified 330 times.

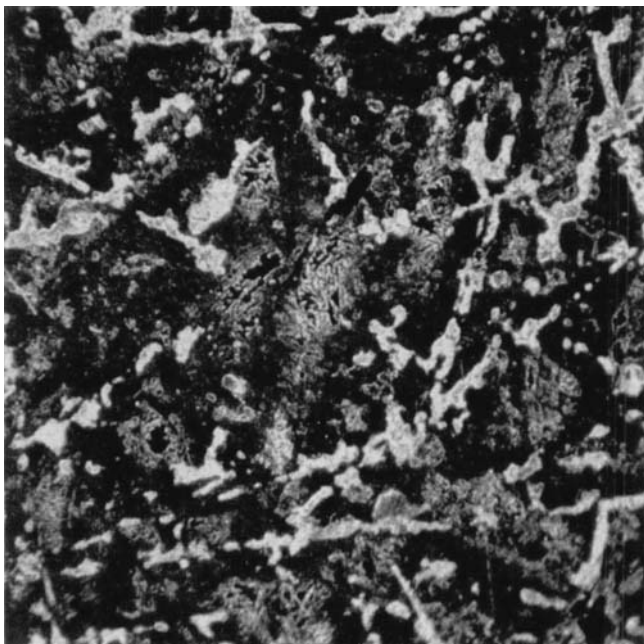


Fig. 2 A.

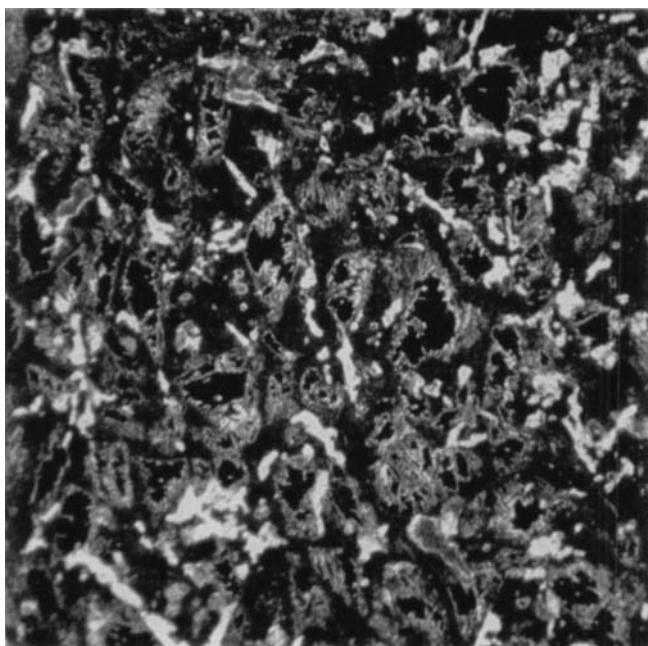


Fig. 2 B.

Figs. 2 A - 2 B. Legend on page 505.

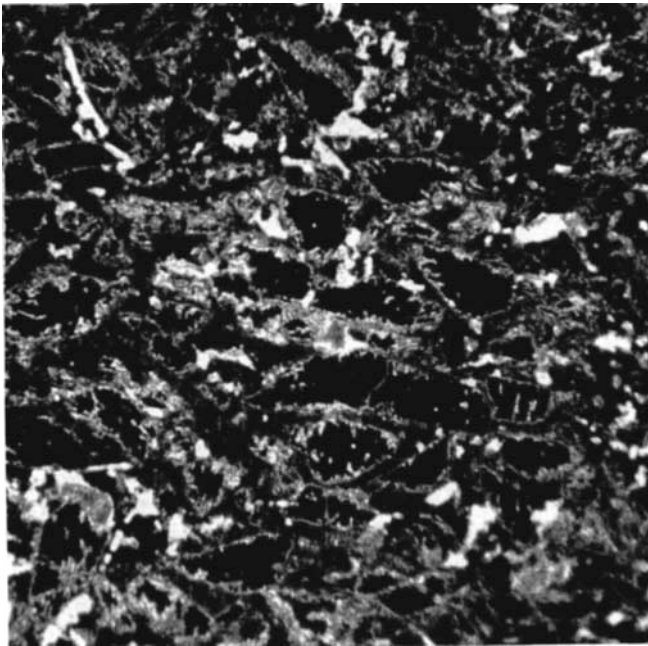


Fig. 2 C.

Fig. 2. Microsections of amalgam with different mercury content due to variation in condensation pressure. A, 54 % Hg. B, 49 % Hg. C, 39 % Hg. Note the increasing amount of deep black γ -grains, and the decreasing amount of large, light grey crystals of the γ_2 -phase. Magnified 280 times.

manufacturers' instructions. The residual mercury content was determined by a method described by *Jørgensen & Nielsen* (1964, p. 230), and gave the results shown in Table I*).

For determination of crushing strength ten specimens for each combination of variables were made from each alloy. After three days the specimens were crushed in a 1000 kg Losenhausen tester with a loading rate of 10 kg per second. The results of these measurements are given in Table II*).

The samples used in the microstructural studies of the amalgams were prepared in the following way: Cylindrical specimens were obtained as described above. Twenty-four hours or more

*) Since only two of the alloy brands were used for complete structural examination, no values are recorded for mercury content and crushing strength of the third brand.

after preparation they were mounted in the self-curing plastic material Palatal P 6 (Badische Anilin- and Soda-Fabrik AG, Ludwigshafen am Rhein). After polymerization of this material the specimens were ground through lengthwise so that the rectangular surfaces measured approximately 5×10 mm. To avoid any appreciable heating of the amalgam the grinding was made cautiously under water. Next, the specimens were polished with a highly diluted suspension of fine silica powder. Grinding and polishing caused no visible change in the amalgam structure under magnifications of about 500 times. Following polishment the specimens were etched with 30 % nitric acid for 10–15 seconds.

The microscopic structure of the amalgams was examined by Leitz Ultropak technique using a conical beam of incident light for illumination of the objects (by ordinary metallographic procedure the amalgam structure was very indistinct). With this technique unreacted alloy particles (the γ -phase) could be seen very clearly as deep black grains against the background of the darker or lighter grey matrix (Figures 1 and 2). The matrix is distinctly made up of two components, one dark grey (γ_1 , chemical compound of silver and mercury), the other light grey (γ_2 , solid solution crystal of tin and mercury). Since all grain boundaries are light grey, the individual grains of the three different phases must have a certain minimum size to permit of identification. Porosities as well, are easy to diagnose by the Ultropak technique.

In order to see whether the etching altered the microstructure of the amalgam the same area of a polished section was photographed before and after etching for 2, 5, 10, and 20 seconds (cf. Figure 1). It was found that structural changes, if any at all, were very slight. Incidentally, it should be mentioned that large, unreacted alloy particles could already be observed in the unetched preparation.

A quantitative analysis of the relative amount of γ -phase was made by means of the Swift Automatic Point Counter (Figure 3), widely used in mineralogy. With this instrument the composition of the preparation is diagnosed in closely and regularly spaced points, 50 μ apart in the present test. Under a magnification of approximately 100, a number of 150–200 points were counted in each of ten different profile lines, i.e. about 1700



Fig. 3. Point counter mounted on the Panphot microscope together with Ultropak objective.

points per preparation. The data, calculated statistically, are presented in Table III. To test the accuracy of the diagnostic procedure the same profile with 100 points was counted independently by both authors with the following results: γ -phase (33,33), matrix (62,63), porosity (5,4), the bracketed figures indicating the two workers' individual findings.

Only one specimen was counted for each combination of variables. Comparison of the counted preparation with at least one control preparation revealed no structural difference.

Table III

Effect of the condensation pressure upon the percentage amount of γ -phase, matrix, and porosity

Pressure, kg	Brand	γ -phase	matrix	pores
10	True Dentalloy	7.6 \pm 2.1	89.9 \pm 3.0	2.5 \pm 1.3
	Solila	7.9 \pm 2.4	91.1 \pm 2.5	1.0 \pm 0.4
30	True Dentalloy	14.4 \pm 2.2	84.5 \pm 2.3	1.1 \pm 0.8
	Solila	14.0 \pm 2.4	84.7 \pm 2.5	1.3 \pm 0.8
60	True Dentalloy	21.9 \pm 3.7	77.3 \pm 3.4	0.8 \pm 0.8
	Solila	20.8 \pm 2.2	78.0 \pm 2.4	1.2 \pm 0.7
120	True Dentalloy	27.8 \pm 2.3	71.0 \pm 2.3	1.2 \pm 0.8
	Solila	27.5 \pm 3.5	71.5 \pm 3.7	0.9 \pm 0.9

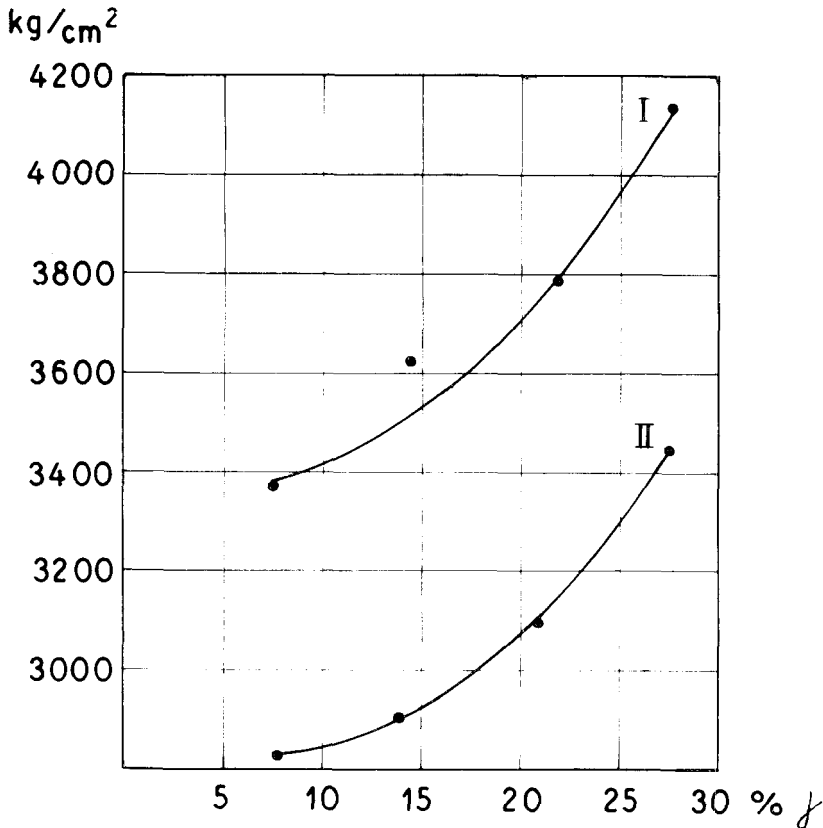


Fig. 4. Percentage amount of residual grains of the γ -phase plotted against crushing strength of the amalgams. I. True Dentalloy. II. Solila Alloy.

Table IV
The influence of mixing time upon amalgam structure

Mixing time, sec.	γ -phase	matrix	pores
10	18.3 ± 1.7	80.7 ± 1.6	1.0 ± 0.2
30	16.7 ± 1.8	82.0 ± 2.0	1.3 ± 0.2
50	17.2 ± 2.8	82.3 ± 3.1	0.5 ± 0.4

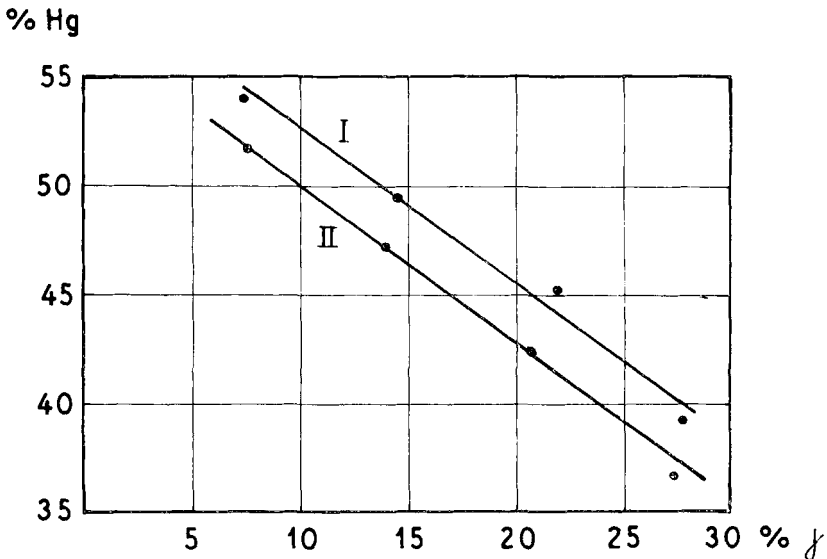


Fig. 5. Percentage amount of residual grains of the γ -phase plotted against residual mercury content. I and II as in Figure 4.

The structure of specimens from DAB Standard alloy was somewhat less clear under the microscope than that of the other two brands. These specimens were therefore not counted; apparently their structure was essentially the same as for the quantitatively tested specimens.

The influence of mixing time upon amalgam structure was examined in a special series of preparations made from True Dentalloy. The results appear in Table IV.

Figures 4 and 5 illustrate the relationship between on one hand the amalgam structure in terms of percentage amount of residual grains (γ -phase), and on the other hand crushing strength and mercury content.

DISCUSSION

It is evident from the present investigation that the amount of γ -phase in silver amalgam can be determined objectively, and that this amount is correlated with such important amalgam properties as crushing strength and mercury content. The point counter method can be used on extremely small surfaces, and in all cases on so small amalgam areas that the material, from a mechanical or physicochemical point of view, no longer can be described as quasi-homogeneous. Thus an amalgam area as Figure 1 A of approximately $1/25$ mm² can be measured quantitatively by repeating the point diagnosis on a suitable number of randomly selected profile lines. The principle of the point counter method can also be utilized without the original apparatus. For example, if a special ocular for grain size determination is used with one half of the ocular field covered with a hexagonal net of lines, the surface of the preparation can be diagnosed in each intersection of the lines. The selection of points will be randomized, and the number of counts in a given area of the preparation can be varied, within wide limits, by varying the magnification of the objective and the mesh size of the hexagonal net.

The method can be used to greatest advantage if the relations shown in Figures 4 and 5 are known. An example is afforded by studies — in progress in this Department for later publication — dealing with the influence of condensation techniques and condensation instruments upon mercury content and crushing strength of amalgam margins.

In practice, an alloy product as e.g. No. I (Fig. 4) can scarcely be condensed to a lower mercury content than 46–48% corresponding to a crushing strength of 3600–3700 kg per cm². It is shown by extrapolation in Figure 4 that amalgam from alloy I with 0% γ -phase has a crushing strength of 3300–3400 kg per cm². The boundary line between γ -containing and γ -free amalgam in a preparation of e.g. an amalgam filling is therefore impor-

tant since it tells us that, relatively, the γ -containing amalgam has a high crushing strength with little difference between the weakest and the strongest areas. A reasonable aim for an acceptable amalgam technique would therefore be fillings without γ -free areas.

SUMMARY

The purpose of the present work is to present a method for a detailed characterization of the structure of amalgam fillings by means of the relative amount of γ -phase, and for showing the relationship between on the one hand the structure and on the other hand the mercury content and the mechanical properties of amalgam. The results are presented in Tables I—III and Figures 2, 4, and 5. The method is useful among other things in studying the influence of types of instruments and technique upon mercury content and mechanical properties of small parts of amalgam fillings.

RÉSUMÉ

ÉTUDES SUR LA STRUCTURE DE L'AMALGAME

I. CORRÉLATION ENTRE LA STRUCTURE ET LES PROPRIÉTÉS PHYSIQUES

Le but du présent travail est de présenter une méthode destinée à caractériser d'une manière détaillée la structure d'obturations d'amalgame au moyen de la quantité relative de phase γ , et à mettre en évidence le rapport entre d'une part la structure et d'autre part la teneur en mercure et les propriétés mécaniques de l'amalgame. Les résultats sont présentés sur les tableaux I—III et les figures 2, 4 et 5. Cette méthode est entre autre utile pour étudier l'influence des différents types d'instruments et de technique sur la teneur en mercure et sur les propriétés mécaniques de petites parties des obturations d'amalgame.

ZUSAMMENFASSUNG

STRUKTURUNTERSUCHUNGEN VON AMALGAM

I. KORRELATION ZWISCHEN STRUKTUR UND PHYSIKALISCHEN EIGENSCHAFTEN

Der Zweck der vorliegenden Arbeit ist die Darlegung einer Methode zur detaillierten Charakteristik der Struktur von Amalgamfüllungen, ausgedrückt durch die relative Menge γ -Phase, so-

wie den Zusammenhang zwischen der Struktur einerseits und dem Quecksilbergehalt und den mechanischen Eigenschaften des Amalgams andererseits zu zeigen. Die Ergebnisse gehen aus den Tabellen I-III und den Figuren 2, 4 und 5 hervor. Die Methode erweist sich als wohlanwendbar, und zwar insbesondere bei Untersuchungen über den Einfluss von Variationen in Instrumenten und Technik auf den Quecksilbergehalt und die mechanischen Eigenschaften der Amalgamkanten.

REFERENCES

- Crawford, W. H. & J. H. Larson*, 1955: Residual mercury determination process. *J. dent. Res.* 34: 313-317.
- Jørgensen, K. D., K. Høst & G. P. Palbøl*, 1964: Sølvamalgams kviksølvyndhold i afhængighed af kondenseringsbegyndelse efter udrøringen. *Tandlægebladet* 68: 3-8.
- Jørgensen, K. D. & M. R. Nielsen*, 1964: Kondenseringsstrykkets indflydelse på brudstyrke og kviksølvyndhold i amalgam. *Tandlægebladet*, 68: 229-234.
- Obst, S.*, 1953: *Ritzhärteprüfungen an Amalgamen unter besonderer Berücksichtigung der praktischen Verarbeitung*. Dissertation. Göttingen.
- Swartz, M. L. & R. W. Phillips*, 1958: Residual mercury content of amalgam restorations and its influence on compressive strength. *The Dental Delineator* 9: 3-8, 18.
- The Swift Automatic Point Counter*. James Swift & Son Limited, 113-115 a Camberwell Road, London S. E. 5, England.