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## **Investigation of some Dental Filling Materials.**

By

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616, 314 × 1.

When planning the new building of the School of Dentistry (Danmarks Tandlægehøjskole) space was reserved for a Chemistry Department. This department should not only have access to an auditorium especially equipped for chemical demonstrations, and to a training laboratory, but should also include working rooms for the chief and the new permanent assistant as well as a room with equipment for testing materials, and a mechanical workshop. The latter should also be at the service of the other departments if the building of apparatus for scientific investigations was required. (See Fig. 1).

These new rooms were occupied at the end of March 1941. During the past year a large part of the time has been spent, besides on instruction, on the furnishing of the rooms, arrangement of the collections, and the development of equipment for material testing. The latter part of the functions of the laboratory had to be built completely from the ground, and conditions necessitated, moreover, that a substantial part of the instruments had to be built, even designed, in the mechanical workshop of the school.

It should be evident that a plan so broad in shape could not be realized in the course of fifteen months. There still lacks a number of instruments, and for years there will undoubtedly be the need of still more instruments for testing of materials, equipment which must be purchased or built in the workshop.

For these reasons the laboratory is at present unable to carry out a complete investigation of materials, which covers all necessary determinations of chemical, physical and mechanical prop-

erties of the material in question. When nevertheless we ventured — at the request of Professor J. J. HOLST — to undertake an investigation of some dental filling materials as soon as possible, *i. e.*, in as early as in the beginning of the fall of 1941, it was with the full knowledge that the result was bound to be incomplete. But even an incomplete investigation may be of

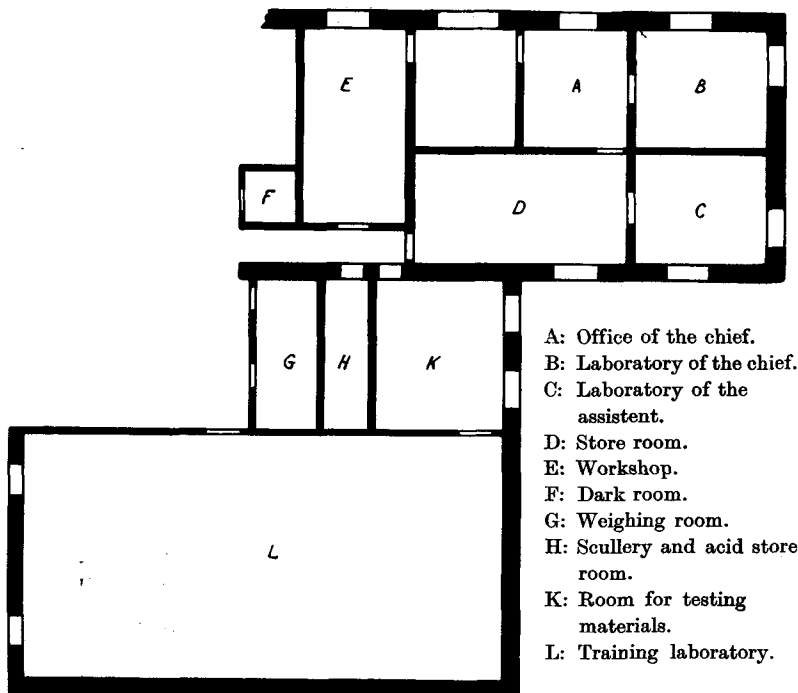


Fig. 1.

some value, and as conditions are at present, prompt information is perhaps more valuable than information late in arriving, even when the latter data are more complete. At the same time this investigation afforded an opportunity to train the staff, gain some experience, and test the available equipment. The result is that we now have a series of temporary measurements of a small number of materials, and that, at the same time, we are better equipped and prepared than a year ago, so that the investigations now in progress can be carried through more rapidly and more completely than our first ones.

The materials investigated were Acolit, Randolf, Silca, Pontural, Dentacryl and Dentex. The following determinations were made: Composition, resistance to certain aqueous solutions, (chemical corrosion), resistance to wear, tensile strength, transverse strength, hardness, compressive strength, and flow.

### 1. Composition.

*Acolit.* According to information furnished by Professor CHR. HOLST this alloy has the composition:

|                 |                 |
|-----------------|-----------------|
| Tin .....       | 78.25 per cent. |
| Antimony .....  | 16.57 » »       |
| Cadmium .....   | 5.13 » »        |
| Aluminium ..... | 0.05 » »        |

The other alloys have been analysed by ourselves with the following results:

#### *Randolf*

|              |                 |
|--------------|-----------------|
| Copper ..... | 70.67 per cent. |
| Zinc .....   | 29.28 » »       |

#### *Silca*

|               |                 |
|---------------|-----------------|
| Silver .....  | 79.03 per cent. |
| Tin .....     | 10.27 » »       |
| Cadmium ..... | 10.54 » »       |

#### *Pontural*

|                 |               |
|-----------------|---------------|
| Aluminium ..... | 94.4 per cent |
| Magnesium ..... | 5.5 » »       |

In the case of *Dentacryl* we have determined the equivalent weight of the liquid which after mixing with the accompanying powder and heating forms the finished product. Saponifying weighed samples of the liquid with alcoholic sodium hydroxide and titrating back the excess of base, led in two determinations to the equivalent weights of 96.3 and 96.4. The formula for the methyl ester of methacrylic acid,  $\text{CH}_2 = \text{C}(\text{CH}_3) \text{COOCH}_3$ , gives the equivalent weight of 100.0. Thus it appears that the liquid, under the reasonable assumption that it mainly consists of the ester in question, contains a few per cent of other substances, presumably negative catalysts which are to inhibit the spontaneous polymerization of the liquid.

## 2. Corrosion.

The experiments were carried out in flat-bottomed test tubes having a height of 10 cm and a diameter of 2 cm. Each test tube was equipped with a rubber stopper with 3 holes (see fig. 2). One hole held a bent glass tube A, bent upward at the bottom end. It had an outside diameter of 5 mm and was drawn out to an opening of about 1 mm. The other hole contained a solid glass rod B which at the bottom end was bent upward to form a hook. The third hole held a bent glass tube C which terminated just below the stopper.

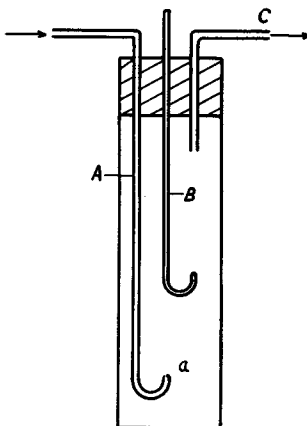


Fig. 2.

From the alloys investigated test specimens of the dimensions  $10 \times 10 \times 2.5$  mm were prepared by the usual centrifugal casting method. The test specimens of Dentacryl were furnished by the firm "Exoglan". A number was stamped into each specimen, and in one corner a hole was drilled, having a diameter of 1.5 mm. A loop of sewing thread was run through this hole, making it possible to hang the test specimen on the hook B. This hook was turned so that the specimen in question hung vertically above the opening a. Then  $\frac{2}{3}$  of the test tube was filled with the corrosion fluid.

The solutions used were, according to WEIKART (1):

I. An aqueous solution containing 1 per cent of lactic acid and 0.25 per cent of sodium chloride. The  $p_H$ -value, measured electrometrically, was found to be 2.28.

II. A 0.5 per cent solution of sodium bicarbonate in water.  
 $p_H = 8.56$ .

$p_H$  was measured by means of a valve potentiometer PHM11, from the firm "Radiometer".

As fig. 3 shows the 12 test tubes were arranged in a row. V is a bottle with water. Solution I was filled into Nos. 1 to 5, and solution II into Nos. 8 to 12. Nos. 6 and 7 were empty and served to catch fluid drops which the air might possibly carry along from No. 5. The whole aggregate was placed in an air thermostat at 37° C. This temperature was chosen to approximate actual conditions as closely as possible. Compressed air was then led through the wash bottle and on through the test tubes.

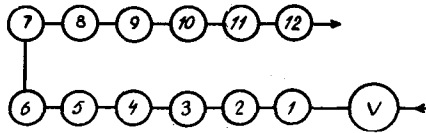


Fig. 3.

The test specimens were weighed before the experiment and after air had been led through for 14 days. Prior to the last weighing they were rinsed in distilled water and dried in open air to constant weight. As it will be seen from the following, some reservations must be taken with respect to the weight of the dentacryl samples found in this way.

The test specimens were then placed in fresh solutions of the same composition as before, and once more treated with air for 14 days. Following drying and weighing the solutions were again renewed and the treatment continued for an additional 28 days.

Table I shows the weight losses in milligram per  $cm^2$  of (original) surface.

Table I.

|                     | Sol. I. |      |       | Sol. II. |     |     |
|---------------------|---------|------|-------|----------|-----|-----|
|                     | 14      | 28   | 56    | 14       | 28  | 56  |
| Days . . . . .      | 14      | 28   | 56    | 14       | 28  | 56  |
| Acolit . . . . .    | 31.6    | 36.1 | 44.7  | 0.8      | 1.6 | 2.2 |
| Randolf . . . . .   | 29.4    | 59.4 | 116.4 | 0.1      | 0.1 | 0.2 |
| Silca . . . . .     | 0.5     | 0.5  | 1.4   | 0.2      | 0.4 | 0.5 |
| Pontural . . . . .  | 22.0    | 52.7 | 107.2 | 0.1      | 0.2 | 0.5 |
| Dentacryl . . . . . | 0.0     | 0.7  | —     | 0.0      | 0.6 | —   |

Fig. 4, which illustrates the conditions in the case of solution I, seems to show that the corrosion of Randolph, Pontural and Silca is proportional with the time, while Acolit in the beginning is very strongly attacked whereupon the relation between corrosion and time becomes linear.

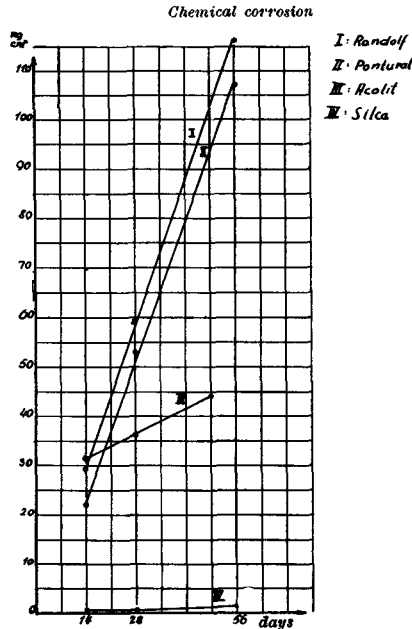


Fig. 4. Chemical corrosion.

The test specimens and the solutions suffered the following changes during the experiment:

*Acolit* in solution I became coated with a grayish-yellow layer. The solution became turbid, and at the bottom there was a white precipitate which at any rate contained tin and probably consisted of stannic acid. In solution 2 the specimen became coated with a black layer, but the solution remained clear.

*Randolf* in solution I became coated with a black layer, while the solution remained clear. In solution II the test specimen showed a brown-colored tarnish and the solution turned slightly turbid.

*Silca* in solution I became coated with a black, loose layer. The solution remained clear. In solution II the test specimen was

slightly tarnished, developing a faintly bronze-like color. Here too the solution was clear.

*Pontural* in solution I developed a porous surface. There was no precipitate in the solution. In solution II the surface lost its metal lustre and the solution became turbid.

*Dentacryl* was apparently unchanged in both solutions, and these remained perfectly clear.

These observations confirm what has previously been known, namely that the phenomenon of corrosion is extremely complex and presents some very strange problems. When a fixed or loose layer is formed on the test specimen, while at the same time precipitate or turbidity occurs in the solution (Acolit I, Randolph II, Pontural II), it is evidently completely impossible, without a closer analysis of the newly formed layer and of the precipitate or the turbidity, to gain any knowledge whatever of what has occurred or how much the material itself really has lost. Such analyses would, at best, require far more work than we have been able to devote to this particular question.

In some instances (Acolit II, Randolph I, Silca I) the test specimen became covered with a layer while the solution remained absolutely clear and apparently unchanged. Though in these instances one should expect an increase in weight, caused by the formation of the new layer, a weight loss was found in all three instances, which, in case of Randolph, even reached the highest of all values found. The most obvious possibility, that soluble salts had been formed — in the case of Randolph primary zinc salts — could not be verified. The question must therefore remain an open one for the time being.

From these observations it is apparent that the corrosion loss found must be accepted with considerable reservation, and that the beautiful linear course of the curves in fig. 4 only means that the test specimens have been treated identically at the three sets of weighings.

In the case of Dentacryl the weight loss was so small in both solutions that it was without significance. Since the possibility existed that eventual losses in weight might be hidden by a simultaneous uptake of water, the following experiments were made.

### 3. The Reaction of Dentaeryl to water.

Two cylindrical test specimens were dried to constant weight over concentrated sulphuric acid. Length and diameter were measured by means of a micrometer caliper. The specimens were then placed in distilled water at 37°. At suitable intervals the rods were picked up, dried, measured and weighed, until the weight, after 4 weeks, became constant. The result are shown in table 2.

Both the expansion and the increase in weight are thus measurable though rather small, and very slow in developing. The tests on resistance to wear, mentioned below, show that also the drying of Dentaeryl to constant weight is a very slow process, so that exact measurements of this material present considerable difficulties.

**Table 2.**

| Sample       | Length in mm              |        |       | Diameter in mm |             |        |        |      |
|--------------|---------------------------|--------|-------|----------------|-------------|--------|--------|------|
|              | before                    | after  | Diff. | before         | after       | Diff.  |        |      |
| I . . . . .  | 20.385                    | 20.462 | 0.077 | 4.874          | 4.893       | 0.019  |        |      |
| II . . . . . | 20.316                    | 20.400 | 0.084 | 4.872          | 4.890       | 0.018  |        |      |
| Sample       | Volume in mm <sup>3</sup> |        |       |                | Weight in g |        |        |      |
|              | before                    | after  | Diff. | pCt.           | before      | after  | Diff.  | pCt. |
| I . . . . .  | 380.0                     | 384.7  | 4.7   | 1.2            | 0.4511      | 0.4582 | 0.0063 | 1.4  |
| II . . . . . | 378.8                     | 383.0  | 4.2   | 1.1            | 0.4495      | 0.4554 | 0.0059 | 1.3  |

### 4. Resistance to wear.

In measurements of this type we considered it quite essential that conditions should, as closely as possible, correspond to those to which the materials are exposed in the mouth cavity. After a critical survey of the available methods we decided to develop further a very simple apparatus mentioned by J. J. HOLST and E. RANCHE-MADSEN (2) in their investigations of tooth paste, as known from a Pepsodent pamphlet.

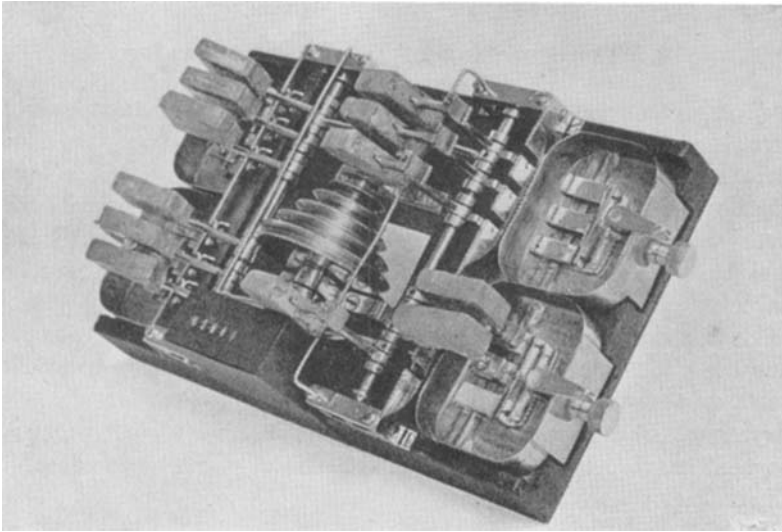


Fig. 5.

Fig. 5 shows the complete arrangement, fig. 6 the schematic construction of one unit. The object was to investigate the resistance to wear of each individual material, partly when wearing against a piece of the same material, and partly when wearing against the other materials in turn. In order to eliminate, if possible, systematic errors in the set-up it was also decided to test each specimen in fixed position as well as in motion.

For this purpose two sets of test specimens were prepared from each material. The fixed ones, designated as *u*, had the dimensions  $10 \times 20 \times 2.5$  mm — the movable, called *o*, the dimensions  $10 \times 10 \times 2.5$  mm. All of them, except the Dentacryl pieces, were made by centrifugal casting.

By means of end-screws the piece *u* is fastened in a holder, attached to the bottom of the metal dish *A* (see fig. 6). The piece *o* is, likewise by means of screws, fastened in a holder which is

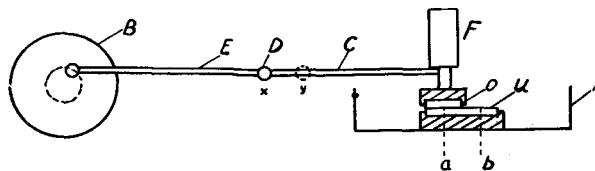


Fig. 6.

held down by a piece of lead F weighing 100 g., in order to keep the wearing surfaces of the two pieces in contact under constant pressure. The holder of the test piece o is fastened to the rod C, the other end of which is connected to the horizontal axis D in such a way that C and its attachments can turn about this axis. One end of the connecting rod E is likewise turning freely on the axis D while the other end is attached, eccentrically, to the disc B. The disc can be rotated by means of a round belt drive from a motor, and the number of revolutions per minute can be changed to suit the purpose by means of the numerous transmission discs seen in fig. 5. For each revolution of the disc B, as registered on a counter (fig. 5), the rod D has moved the distance x—y—x (fig. 6), and at the same time the test piece o has moved the distance a—b—a, which is 20 mm. Thus the upper test piece contacts the lower one with its whole bottom surface during the entire movement. Each test series comprised  $5 \cdot 10^6$  revolutions, causing the two test pieces to pass each other a total of 1 million times. This figure was chosen on the assumption that a person normally masticates about 25 times for each mouthful. If we estimate a sandwich (Danish) at 6 mouthfuls, an average lunch will require, roundly, 1,000 masticatory movements, thus one day's eating 3,000 movements at the most.  $10^6$  frictional movements should therefore correspond to about 1 year's mastication.

As fig. 5 shows, the whole apparatus contains 12 units like the one described, divided into 4 groups of 3 each, with a special dish for each group. In each group of 3 units the upper test pieces are identical, and the lower ones likewise identical with one another. Thus it is possible with this set-up to test simultaneously 4 of the 25 possible combinations.

The following solutions were used:

I. A mixture of 25 per cent glycerin and 75 per cent water. By means of Ostwald's viscosimeter the viscosity was found to be 2.1 at room temperature. As a rule, the viscosity of saliva is stated to be about 2.

II. An artificial saliva<sup>1</sup> consisting of

|                                   |       |
|-----------------------------------|-------|
| Potassium chloride . . . . .      | 2.4 g |
| Calcium phosphate (tri) . . . . . | 0.6 g |
| Potassium sulphate . . . . .      | 0.9 g |

<sup>1</sup> Journ. amer. dental. assoc. 1941, No. 8, p. 1284.

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|                                |         |
|--------------------------------|---------|
| Sec. potassium phosphate ..... | 1.4 g   |
| Norm. sodium phosphate .....   | 0.8 g   |
| Albumin .....                  | 5.0 g   |
| Water .....                    | 1,000 g |

By electrometric measurement the mixture was found to have a pH-value of 9.33. Thus it is strongly basic.

Before and after each experiment the test specimens were rinsed, dried off, dried in desiccator, and weighed.

In spite of all care we did not succeed, however, in obtaining reproducible results by this method. For one thing, the differences between the values of the resistance to wear found for the individual material, when placed above or below was far too great in view of the fact that the pressure and the wearing surface were the same for the upper and the lower test piece.

Table 3 shows the mean values of all measurements, 6 in case of each pair of materials. A denotes Acolit, R Randolf, S Silca, P Pontural and D Dentacryl. The losses are given in milligram.

Table 3.

| Solutions: |             | I      | II                  |
|------------|-------------|--------|---------------------|
| A          | 2,2 against | A 2.2  | A 2.0 against A 2.0 |
| A          | 2.5 ›       | R 0.9  | A 2.4 › R 0.5       |
| A          | 1.5 ›       | S 0.6  | A 1.7 › S 0.6       |
| A          | 4.0 ›       | P 15.7 | A 3.2 › P 0.6       |
| A          | 0.3 ›       | D —    | A 0.7 › D 1.1       |
| R          | 0 ›         | R 0    | R 0 › R 0           |
| R          | 0.4 ›       | S 0.5  | R 0.1 › S 0.9       |
| R          | 4.7 ›       | P 18.4 | R — › P —           |
| R          | — ›         | D —    | R 0.7 › D 0.5       |
| S          | 1.2 ›       | S 1.2  | S 1.9 › S 1.9       |
| S          | 3.0 ›       | P 1.5  | S — › P —           |
| S          | 1.2 ›       | D 1.8  | S 1.5 › D 0.2       |
| P          | 81.7 ›      | P 81.7 | P 183 › P 183       |
| D          | 3.4 ›       | D 3.4  | D 0.1 › D 0.1       |

The table shows that the resistance to wear of a material to a high degree depends on what other material it is brought into contact with, and, next, on the nature and composition of the

liquid. Thus it is found in the case of Pontural that its loss on wear in solution II varies from 0.6 to 182 mg, all according to whether the opposite abrading piece is Acolit or Pontural. In solution I, however, the corresponding values are 15.7 and 81.7 mg respectively. Disregarding the low values against Acolit (solution II) and Silca (solution I) it is found that the Pontural is far less resistant to wear than the other materials investigated. The values for Acolit varies from 0.3 to 4.0 mg, for Randolf from 0 to 0.9 mg, for Silca from 0.5 to 3.0 mg, and for Dentacryl — in so far as it was possible at all to obtain constant values at the drying — from 0.1 to 3.4 mg. Thus it appears that of the 5 materials investigated Randolf has the highest, Pontural (as a rule) the lowest resistance to wear, while the three other materials are about equal.

We have not been able to find any relation whatever between the losses measured and the chemical or electrochemical properties of the materials.

According to the results of these experiments no particular value can be attached to measurements of the resistance to wear in which a single grinding agent has been used, common for all the materials tested. This applies, for example, to BERTIL EKENSTEIN'S (3) investigations of synthetic prosthesis materials where a disc of the material or an ordinary tooth revolved in carborundum powder. The order of the series of values given might possibly have been entirely different if another abrasive had been employed.

### 5. Tensile Strength.

In the determinations of tensile strength a testing machine was used which was designed and built at the Technology Laboratory of the Danish Technical Highschool. It is shown complete in fig. 7 while figs. 8 and 9 give the most important details.

The cylindrical test specimens were cast in lengths of 40 mm and had a diameter of 2.5 mm. They were prepared in part by centrifugal casting and partly by casting in vacuum. The latter process was kindly carried out by the firm of W. Hellesen and Co., while the specimens of Dentacryl were prepared by the firm of Exoglan.

All test specimens were turned down to a diameter of 2.0 mm and then fastened in the testing machine, as shown in fig. 8, in such a way that the free part of the specimen, which was exposed

to the tension, had a length of 20 mm. The motor was then started, causing a *gradually increasing stress*.

The movement of the jaws holding the test specimen being stretched is transferred, by means of a round belt drive, to a recording drum (fig. 9) the turning of which registers the elonga-

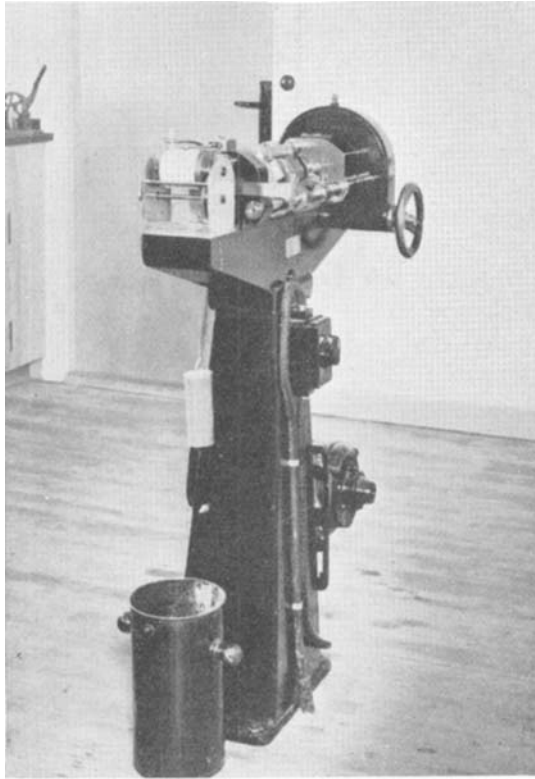


Fig. 7.

tion of the specimen. The conversion factor is 22.5. The acting force is also recorded by means of a heavy spring, the deformation of which is transferred to a long writing pen (fig. 9) by means of a system of levers, and is magnified about 18 times. Hence, on the strip of paper from the recording drum there is obtained a curve, the maximal height of which gives the force exerted at the moment of fracture of the test specimen, thus the tensile strength, while the corresponding elongation is measured on the

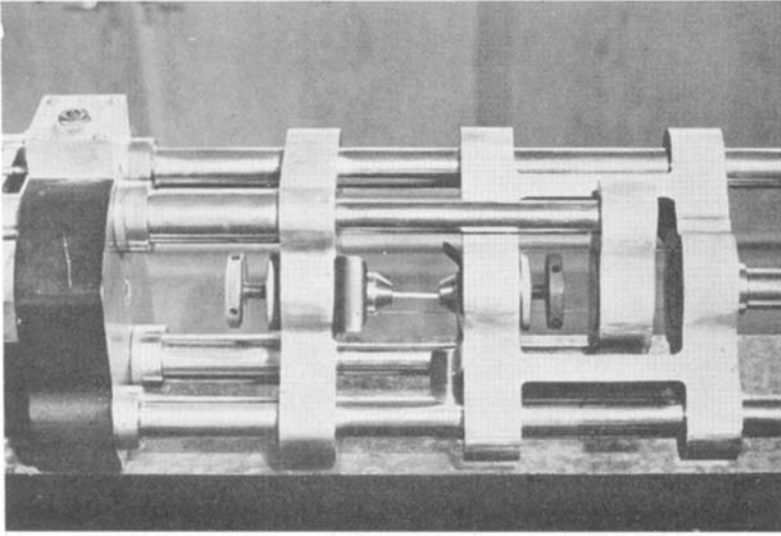


Fig. 8.

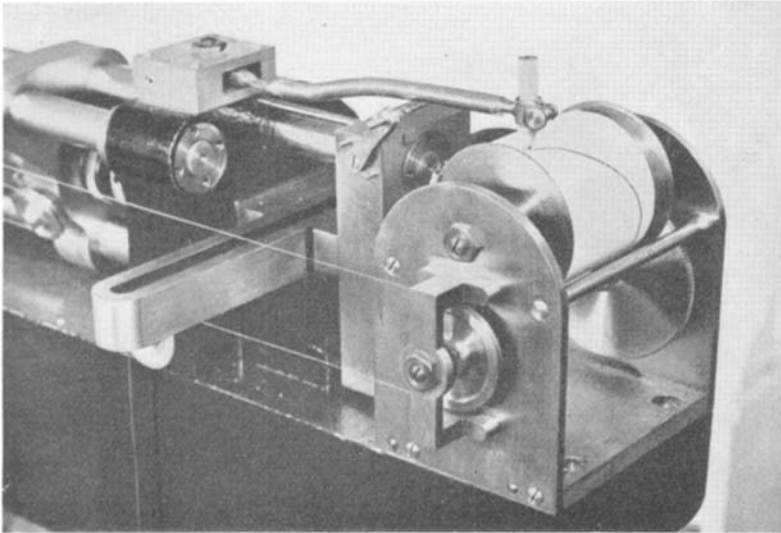


Fig. 9.

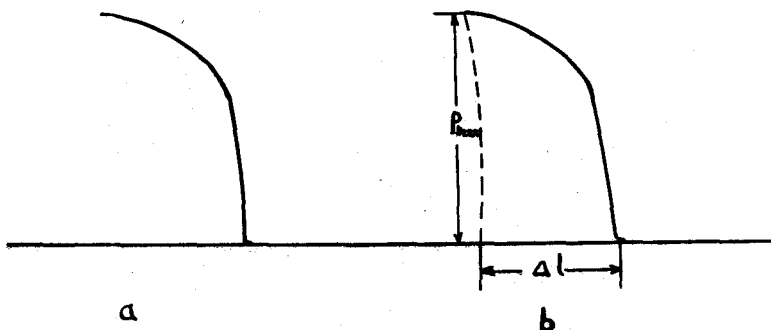


Fig. 10.

base line. Fig. 10 a shows such a curve. Starting from the maximal point a dotted curve is drawn (fig. 10 b), by the aid of a templet, representing the curve described by the writing pen when moving about the other end of the recording arm as center. The elongation,  $\Delta l$ , is measured as the distance from the intersection of this line with the base line to the foot of the curve.

If the maximum tensile stress is  $P_t$  kg, and  $d$  the original diameter of the test specimen (in cm), the tensile strength is found to be

$$\frac{P_t}{\pi \cdot \frac{d^2}{4}} \text{ kg/cm}^2.$$

The results were as follows:

**Table 4.**  
*Centrifugal casting.*

|                    | $P_t$<br>in kg | Tensile<br>strength<br>in kg/cm <sup>2</sup> | Mean | $\Delta l$<br>in mm | Mean |
|--------------------|----------------|--|------|---------------------|------|
| Acolit . . . . .   | 65             | 2070   | 1830 | 0.7                 | 0.5  |
|                    | 50             | 1590   |      | 0.3                 |      |
| Randolf . . . . .  | 80             | 2550   | 2810 | 2.0                 | 1.8  |
|                    | 74             | 2360   |      | 1.0                 |      |
|                    | 110            | 3500   |      | 2.5                 |      |
| Silca . . . . .    | 60             | 1910   | 1590 | 1.1                 | 0.6  |
|                    | 40             | 1270   |      | 0.2                 |      |
| Pontural . . . . . | 40             | 1270   | 1300 | 0.2                 | 0.25 |
|                    | 42             | 1340   |      | 0.3                 |      |

*Casting in vacuo.*

|                    | $P_t$<br>in kg | Tensile<br>strength<br>in $\text{kg}/\text{cm}^2$ | Mean | $\Delta l$<br>in mm | Mean |
|--------------------|----------------|---|------|---------------------|------|
| Acolit . . . . .   | 40             | 1270  | 1390 | 0.3                 | 0.3  |
|                    | 47             | 1500  |      | 0.2                 |      |
|                    | 44             | 1400  |      | 0.5                 |      |
| Silca . . . . .    | 46             | 1470  | 1530 | 0.9                 | 1.0  |
|                    | 50             | 1590  |      | 0.8                 |      |
|                    | 48             | 1530  |      | 1.2                 |      |
| Pontural . . . . . | 55             | 1750  | 1700 | 0.4                 | 0.3  |
|                    | 40             | 1590  |      | 0.3                 |      |
|                    | 56             | 1780  |      | 0.3                 |      |

*Dentacryl.*

|  | $P_t$<br>in kg | Tensile<br>strength<br>in $\text{kg}/\text{cm}^2$ | Mean | $\Delta l$<br>in mm | Mean |
|--|----------------|---|------|---------------------|------|
|  | 18             | 575   | 480  | 0.8                 | 0.6  |
|  | 12             | 383   |      | 0.4                 |      |

*Dentex.*

|  | $P_t$<br>in kg | Tensile<br>strength<br>in $\text{kg}/\text{cm}^2$ | Mean | $\Delta l$<br>in mm | Mean |
|--|----------------|---|------|---------------------|------|
|  | 15             | 480   | 500  | 0.6                 | 0.6  |
|  | 15             | 480   |      | 0.6                 |      |
|  | 17             | 550   |      | 0.8                 |      |
|  | 15             | 480   |      | 0.5                 |      |

The vacuum cast test specimen of Randolf were not included since they were so porous and heterogeneous that it was impossible to obtain reasonably reproducible values. As far as the other alloys are concerned, a comparison shows that the vacuum casting has given the highest value in the case of Pontural, while Acolit becomes stronger when cast centrifugally, and Silca, with respect to tensile strength, is independent of the method of casting.

The two measurements of Dentacryl gave values of 383 and 575, with a mean of 480  $\text{kg}/\text{cm}^2$ , while B. KONSTANTIN-HANSEN (4), using a somewhat more primitive procedure, found 385

kg/cm<sup>2</sup> as the value for Exo-Metacryl, a result which checks very well with our lowest value.

The considerable deviations found among the individual measurements especially of the elongation  $\Delta l$ , must mainly be ascribed to the heterogeneity of the material, whether this defect is inherent or is caused by the treatment, especially the casting of the test specimens.

## 6. Transverse Strength.

The measurements were made by means of the apparatus shown in fig. 11, furnished by Louis Schopper, Leipzig. The piece L, which rests on the pressure plate A, can be raised by means of a spindle B towards the cylinder H, the diameter of

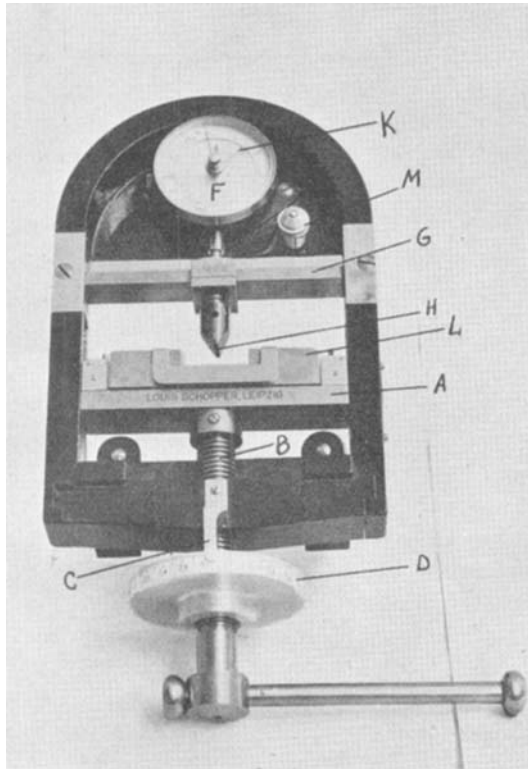


Fig. 11.

which is 3.2 mm. (see fig. 12). The movements of the spindle is measured on the scale C and the measuring drum C. The test specimen is placed on L so that it just touches H, but without causing any deflection of the hand on the measuring dial F. Then the screw which fastens the drum D on the spindle is loosened, and the drum is moved and turned until its zero-point coincides with the zero-point on the scale C. The drum is then fastened by means of the screw.

The dial has, besides the hand proper which records the pressure, an auxiliary hand which is turned to zero position.

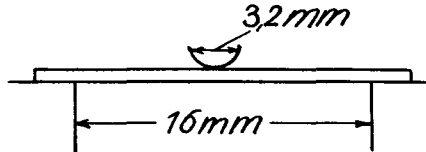


Fig. 12.

When making measurement the handle of the spindle is first turned to the right at a rate as uniform as possible until the test specimen breaks, whereupon the turning is stopped immediately. The auxiliary hand will then show the maximum pressure in kilograms ( $P_b$ ) on the outer scale of the measuring dial. At the same time as the test specimen is bent downward, the spring G is pressed upward, and its displacement is read on the inner scale of the dial. The actual bending is then determined as the difference between the recordings of the measuring drum and the measuring dial.

The test specimens were cylinders with a diameter of 5 mm and a length of 22 mm. Except for the Dentacryl sample, which was furnished by the firm of Exoglan, all were prepared by centrifugal casting.

The transverse strength is calculated from the formula

$$\sigma = \frac{8 \cdot l}{\pi \cdot d^3} \cdot P_b,$$

where  $P_b$  is the maximum pressure in kg.,  $l$  the distance between the points of support in cm = 1.6 cm (fig. 12) and  $d$  the diameter of the test specimen in cm = 0.5 cm.

Since the latter two quantities are the same for all the samples we find

$$\sigma = 32.60 P_b \text{ kg/cm}^2.$$

The performance of this test was only possible in the cases of Acolit, Pontural Dentacryl and Dentex, since both Randolf and Silca could be bent without breaking, even after the force had been increased to 250 kg, which was the maximum for the apparatus Table 5 shows the results.

The values for Dentacryl and Dentex are in very poor agreement with the value found by B. KONSTANTIN-HANSEN (5) in case of Exo-Metacryl — 1,160 kg/cm<sup>2</sup>. In KONSTANTIN-HANSEN's measurements the test specimens — having a diameter of 0.3 cm and a free length of 5 or 3 cm — were charged with a load at the center consisting of a tin container which was filled with lead shot until the specimen broke. Since Dentacryl (see later) shows an appreciable flow, it is possible that the speed with which the load is applied strongly influences the transverse strength.

Table 5.

|                     | P <sub>b</sub> in kg | Transverse strength | Mean |
|---------------------|----------------------|---------------------|------|
| Acolit . . . . .    | 65                   | 2120                | 2390 |
|                     | 70                   | 2280                |      |
|                     | 85                   | 2770                |      |
| Pontural . . . . .  | 67                   | 2180                | 2230 |
|                     | 70                   | 2280                |      |
| Dentacryl . . . . . | 15.0                 | 490                 | 450  |
|                     | 12.5                 | 408                 |      |
|                     | 14.0                 | 456                 |      |
| Dentex . . . . .    | 12                   | 390                 | 530  |
|                     | 30                   | 980                 |      |
|                     | 25                   | 815                 |      |
|                     | 14                   | 460                 |      |
|                     | 27                   | 880                 |      |
|                     | 13                   | 420                 |      |
|                     | 20                   | 650                 |      |
|                     | 15                   | 490                 |      |
|                     | 23                   | 750                 |      |
|                     | 10                   | 330                 |      |
|                     | 7                    | 230                 |      |
|                     | 13                   | 420                 |      |
|                     | 13                   | 420                 |      |
| 13                  | 420                  |                     |      |
| 7                   | 230                  |                     |      |

This question will be further investigated, but at present it is hardly possible to compare results obtained by so different methods.

### 7. Hardness.

When measuring the hardness the same apparatus was used as in the measurement of transverse strength, with the difference that the cylinder H was replaced by a Brinell ball M, having a diameter of 5 mm. Moreover, the piece L was removed.

The test specimens were plates with two plano-parallel surfaces. The test specimen was placed on the pressure plate A which was adjusted so that the specimen just touched the Brinell ball. Then the measuring drum D was adjusted as in the measurement of transverse strength.

The measurement is performed by turning the spindle until the hand of the dial stands at 50 kg. The depth to which the ball is forced into the test specimen is then found as the difference between the readings on the measuring drum and the dial (see the foregoing section).

The Brinell hardness is calculated from the formula

$$H = \frac{P}{\pi \cdot D \cdot t} \text{ kg/cm}^2,$$

where P is the pressure applied = 50 kg. D is the diameter of the ball = 0.5 cm, and t the depth of the impression in cm.

Hence

$$H = \frac{50}{\pi \cdot 0.5 \cdot t} = \frac{31.9}{t} \text{ kg/cm}^2.$$

10 measurements were made in case of each of the four alloys. Table 6 gives the depth of the impression in cm.

Instead of measuring the depth of the impression one may also measure its diameter. Provided the material is not elastic the two methods must necessarily lead to the same result. This is true, for example, of the alloys investigated. Dentacryl, however, possesses a measurable elasticity which results in a reduction of the depth of the impression when the pressure is released, so that the diameter of the impression no longer corresponds to the depth found under pressure. Since we intend to start an investigation of the properties of different prosthesis materials, including measurements of their elasticity and plasticity we consider it better to postpone the investigation of Dentacryl till then, and refrain from discussing the question any further here.

Table 6.

|                  | Acolit | Randolf | Silca  | Pontural                |
|------------------|--------|---------|--------|-------------------------|
|                  | 0.0150 | 0.0105  | 0.0110 | 0.0160                  |
|                  | 0.0120 | 0.0120  | 0.0130 | 0.0150                  |
|                  | 0.0143 | 0.0110  | 0.0100 | 0.0152                  |
|                  | 0.0140 | 0.0105  | 0.0100 | 0.0160                  |
|                  | 0.0112 | 0.0121  | 0.0120 | 0.0150                  |
|                  | 0.0140 | 0.0100  | 0.0100 | 0.0150                  |
|                  | 0.0120 | 0.0100  | 0.0100 | 0.0140                  |
|                  | 0.0135 | 0.0095  | 0.0112 | 0.0150                  |
|                  | 0.0130 | 0.0100  | 0.0118 | 0.0155                  |
|                  | 0.0130 | 0.0104  | 0.0100 | 0.0140                  |
| Mean             | 0.0134 | 0.0106  | 0.0109 | 0.0151                  |
| Brinell-hardness | 2400   | 3010    | 2926   | 2116 kg/cm <sup>2</sup> |

### 8. Compressive Strength.

In the above mentioned tensile testing machine the direction of movement can be reversed, so that the two jaws approach each other at constant speed. If both jaws are replaced by cylindrical pieces of steel with solid end surfaces (pressure plates) the machine is directly applicable to the measurement of compressive strength. However, this function of the machine could not be utilized completely in the present investigation, since it was found that none of our five materials could be crushed at the highest pressure the machine was capable of exerting (225 kg).

In our opinion, however, it is of far more interest to measure the *deformation* of the material under pressure. Like most other changes of form produced under the influence of mechanical stresses, the deformation under pressure depends on a series of different properties of the material. These properties are called elasticity, plasticity, toughness, brittleness, hardness and flow, without it being possible always to decide which of these properties, alone or combined with others, is responsible for the changes measured. The flow — which, as a rule, is defined as the deformation (under pressure) per unit of time — introduces the time factor, and the same is sometimes true of the elastic deformations as well. A deformation of one and the same substance may thus be purely elastic when the deformation is rapid, but

purely plastic when it is slow. Hence it is apparent that values for these deformations can only be of significance when it is stated how they are obtained, and that values obtained by different methods cannot, as a rule, be compared. But it may always be of importance to compare values for different materials, determined by means of a given method, provided this method, as far as possible, reproduces the conditions under which the materials are used.

Widely different figures have been given for the pressure exerted in mastication. SKINNER (6) reports a variation from 2,500 to 27,500 lbs., average 17,000 lbs., per square inch. The mean value corresponds to 1,180 kg/cm<sup>2</sup>.

Our test specimens were cylindrical, having a length of 2 mm and a diameter of 2 mm. The maximum pressure applied was 200 kg, which corresponds to 6,350 kg/cm<sup>2</sup>, thus considerably higher than the highest pressure measured during mastication. At the maximum pressure we found the following length reductions,  $\Delta l$ , in mm:

Table 7.

|                     | $\Delta l$ for<br>200 kg     | Mean | $\Delta l$ in<br>pCt. | $\Delta l$ for<br>37 kg | $\Delta l$ in<br>pCt. |
|---------------------|------------------------------|------|-----------------------|-------------------------|-----------------------|
| Acolit . . . . .    | 1.42<br>1.30<br>1.61         | 1.44 | 72                    | 0.07                    | 3.5                   |
| Randolf . . . . .   | 0.71<br>0.55<br>0.72         | 0.66 | 33                    | 0.06                    | 3.0                   |
| Silca . . . . .     | 0.87<br>0.70<br>0.80<br>1.52 | 0.79 | 40                    | 0.06                    | 3.0                   |
| Pontural . . . . .  | 1.52<br>1.50<br>1.65         | 1.56 | 78                    | 0.21                    | 10.5                  |
| Dentacryl . . . . . | 1.40<br>1.41<br>1.48         | 1.43 | 72                    | 0.14                    | 7.0                   |
| Dentex . . . . .    | 1.5<br>1.35<br>1.5           | 1.44 | 77                    | 0.2                     | 10                    |

Thus Acolit, Pontural, Dentacryl and Dentex were completely flattened at a pressure of 200 kg, while Randolf and Silca were better able to resist the pressure.

Fig. 13 shows the relation between the length reduction (mm) and the pressure applied. The average pressure of mastication corresponds to the ordinate 37. The values for  $\Delta l$  given in the last column but one of table 7 are read from the curves at this

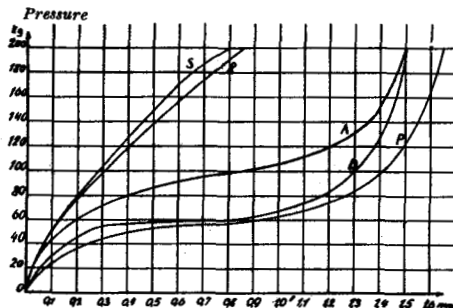


Fig. 13. Pressure.

pressure; in the last column of the table they are converted into per cent. It will be seen that Acolit, Randolf and Silca under these conditions have about the same resistance to pressure, while the strength of Dentacryl is somewhat, and that of Dentex and Pontural considerably lower.

## 9. Flow.

The deformation which occurs under the influence of pressure is frequently — like most of the other changes which are produced by mechanical influences — dependent on the length of the period during which the pressure is exerted. Hence it is necessary to measure also the ratio between the deformation and the time during which the force is applied, *i. e.*, the flow.

Fig. 14 shows the apparatus used. It was designed and built in our mechanical workshop. The steel piston B glides in the cylinder A. C is a pawl which, through a cutout in the cylinder, follows the displacement of the piston and transfers it to the dial D which is graduated from 1 to 50. Each graduation corresponds to a displacement of the piston of 0.01 mm.

The cylindrical test specimen (diameter 2 mm, height 4 mm) is placed on the bottom plate below the piston. A 10 kg weight F is then placed on the plate which forms the top of the piston. The measuring dial is adjusted by means of the regulating screw

G or by turning the face of the graduated dial so that the hand points to zero.

The whole apparatus is placed in an air thermostat at 37—33°. The recording dial is read at suitable intervals.

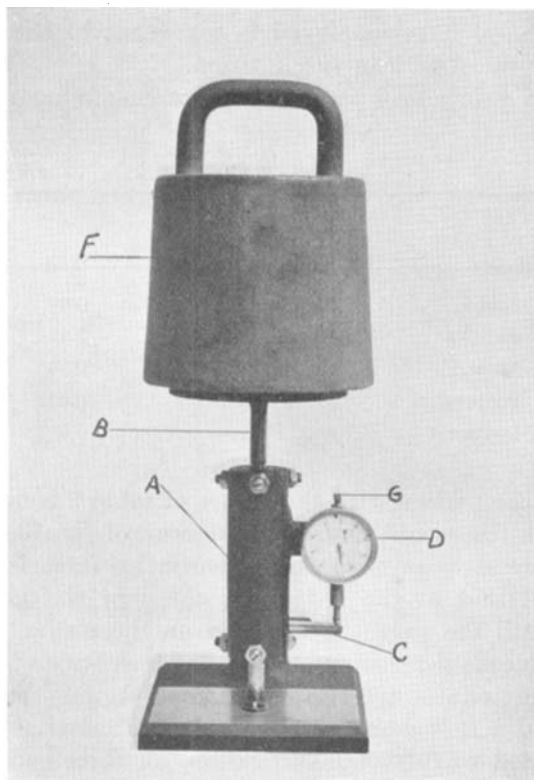


Fig. 14.

As a measure of the flow we have chosen the figure which is recorded on the dial after the force has been applied for 24 hours. Calling this figure  $a$ , the flow, expressed in per cent, is

$$F = \frac{a \cdot 10^{-2}}{4} \cdot 100.$$

The pressure applied is

$$\frac{10}{\pi \cdot r^2} = 320 \text{ kg/cm}^2,$$

thus considerably less than the average masticating pressure. As a comparison may serve that the American Standard (SKINNER, 7) for the measurement of flow calls for a cylindrical test specimen 4 mm in diameter and 8 mm long, a pressure of 250 kg/cm<sup>2</sup>, and a temperature of 20 to 25° C. It will be seen that the pressure we have employed is somewhat higher than that required by the American specification.

The mean values of 3 series of measurements are recorded in table 8.

Table 8.

| F in pCt.           |      |
|---------------------|------|
| Acolit . . . . .    | 0.60 |
| Randolf . . . . .   | 0.02 |
| Silca . . . . .     | 0.00 |
| Pontural . . . . .  | 0.00 |
| Dentacryl . . . . . | 6.0  |

A direct comparison with the values of table 7 is not possible, even though one would find, on the curves of fig. 13, values for the same pressures as in the measurement of flow. It should be remembered that at the latter test the zero position was not adjusted until the weight was placed on the piston, *i. e.*, after an instantaneous deformation might have occurred.

Of the materials investigated, Dentacryl was the only one that showed a flow worth mentioning. This material was therefore subjected to further investigation, all three series of measurements being extended to 2 weeks. Table 9 gives the results, in per cent.

Table 9.

| Sample No.  | 1 hour | 1 day | 2 days | 4 days | 8 days | 14 days |
|-------------|--------|-------|--------|--------|--------|---------|
| 1 . . . . . | 2.0    | 6.6   | 7.6    | 9.1    | 10.6   | 12.2    |
| 2 . . . . . | 1.6    | 6.3   | 7.6    | —      | 11.3   | 13.3    |
| 3 . . . . . | 1.6    | 5.0   | —      | —      | —      | 9.8     |

Fig. 15 shows the course of the deformation the magnitude of which may reach more than 13 per cent of the length in 14

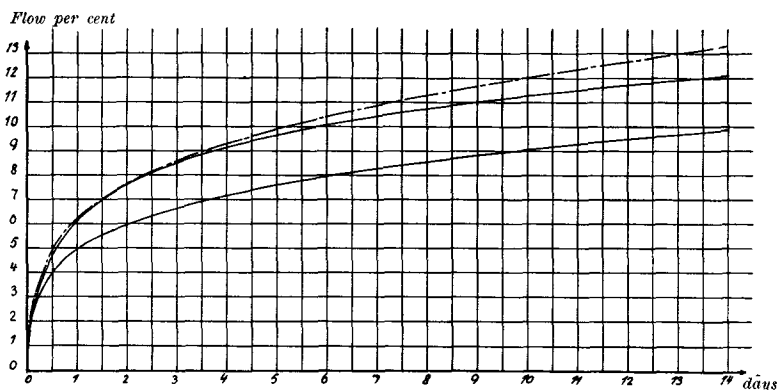


Fig. 15. Flow per cent.

days, apparently without approaching a limit. The only explanation is that the substance flows, and hence must be considered a fluid with a high viscosity.

### 10. Summary of Results.

Table 10 contains all results obtained.

Table 10.

|              | Corrosion in acid solution in 28 days, mg/cm <sup>2</sup> | Appearance after corrosion in acid solution | Tensile strength  |               | Transverse strength in kg/cm <sup>2</sup> | Brinell-hardness | Deformation in per cent at a pressure of 1180 kg/cm <sup>2</sup> | Flow in per cent |
|--------------|---|---|-------------------|---------------|---|------------------|--|------------------|
|              |   |   | Centrifugal cast. | cast. in vac. |   |                  |  |                  |
| Acolit . .   | 36.1  | Discoloring                                 | 1830              | 1390          | 2390                                      | 2400             | 3.5  | 0.6              |
| Randolf . .  | 59.4  | ›   | 2800              | —             | —   | 3010             | 3.0  | 0.02             |
| Silca . . .  | 0.5   | ›   | 1590              | 1530          | —   | 2926             | 3.0  | 0.0              |
| Pontural . . | 52.7  | Porosity                                    | 1300              | 1700          | 2230                                      | 2116             | 10.5   | 0.0              |
| Dentaeryl    | 0.7   | No change                                   | —                 | 480           | 451                                       | —                | 7.0  | 6.0              |

The survey shows that all the alloys investigated, when subjected to corrosion in acid solution became either discolored or porous. On the strength of this test Pontural with its strong deformation under pressure must be considered the poorest of the alloys investigated. Of the three others, Randolf possesses both the highest hardness and tensile strength, and its compress-

sive strength and flow are satisfactory. In contrast to these qualities is the strong corrosion.

Among the four alloys Silca must be considered the most serviceable because of its hardness, compressive strength, low flow, and high resistance to corrosion. Its tensile strength is somewhat lower than that of Randolf, but it is doubtful whether the difference is of significance.

Dentaeryl shows a substantially lower tensile and transverse strength, than the alloys investigated; its flow is considerable. It seems, on the other hand, to be quite resistant to corrosion.

### Zusammenfassung.

Tabelle 10 enthält alle die gefundenen Ergebnisse.

Tabelle 10.

|            | Korrosion<br>in einer<br>Säure<br>während<br>28 Tagen<br>mg/cm <sup>2</sup> | Aussehen<br>nach<br>Korrosion<br>in einer<br>Säure | Dehnungs-<br>festigkeit    |                           | Biege-<br>festig-<br>keit in kg/cm <sup>2</sup> | Brinell-<br>härte | Formveränderung<br>in % unter Druck<br>von 1180 kg/cm <sup>2</sup> | Formveränderung<br>pro Zeiteinheit<br>in % |
|------------|---|--|----------------------------|---------------------------|---|-------------------|--|--|
|            |   |  | Zentri-<br>fugaler<br>Guss | Guss<br>in<br>Va-<br>kuum |   |                   |  |  |
| Acolit . . | 36.6  | Verfärbung   | 1830                       | 1390                      | 2390  | 2400              | 3.5  | 0.6  |
| Randolf .  | 59.4  | „  | 2800                       | —                         | —   | 3010              | 3.0  | 0.02                                       |
| Sika . . . | 0.5   | „  | 1590                       | 1530                      | —   | 2926              | 3.0  | 0.0  |
| Pontural . | 52.7  | Porosität  | 1300                       | 1700                      | 2230  | 2116              | 10.5   | 0.0  |
| Dentaeryl  | 0.7   | Keine<br>Änderung                                  | —                          | 480                       | 451   | —                 | 7.0  | 6.0  |

Die Übersicht zeigt, dass alle die untersuchten Legierungen, wenn sie der zerfressenden Wirkung einer Säure ausgesetzt wurden, sich entweder verfärbten oder porös wurden. Auf Grund dieser Probe ist Pontural mit seiner starker Formveränderung unter Druck als die schlechteste der untersuchten Legierungen anzusehen. Von den drei übrigen besitzt Randolf sowohl die grösste Härte als auch die grösste Dehnungsfestigkeit, und ausserdem sind Druckfestigkeit und Formveränderung pro Zeiteinheit befriedigend. Im Gegensatz zu diesen Eigenschaften steht die starke Korrosion.

Unter den vier Legierungen ist Silca als die zweckmässigste anzusehen wegen ihrer Härte und Druckfestigkeit, ihrer geringen

Formveränderung pro Zeiteinheit und ihrer grossen Widerstandskraft gegen Korrosion. Ihre Dehnungsfestigkeit ist etwas geringer als die von Randolf, doch dürfte zweifelhaft sein, ob der Unterschied irgendwie von Bedeutung ist.

Dentacryl zeigt bedeutend geringere Dehnungs- und Biegungsfestigkeit, als die untersuchten Legierungen, und starke Formveränderung pro Zeiteinheit. Andererseits scheint es völlig korrosionsbeständig zu sein.

### Résumé.

Le tableau 10 embrasse tous les résultats obtenus.

Tableau 10.

|             | Corrosion en solution acide pendant 28 jours mg/cm <sup>2</sup> | Aspect après corrosion en solution acide | Résistance à la tension |              | Résistance à la flexion en kg/cm <sup>2</sup> | Brinell-dureté | Déformation en pourcent sous une pression de 1180 kg/cm <sup>2</sup> | Coulée, en pourcent |
|-------------|---|--|-------------------------|--------------|---|----------------|--|---------------------|
|             |   |  | Fonte centrifugale      | Fonte à vide |   |                |  |                     |
| Acolit . .  | 36.6  | Changement de couleur                    | 1830                    | 1390         | 2390  | 2400           | 3.5  | 0.6                 |
| Randolf . . | 59.4  | »  | 2800                    | —            | —   | 3010           | 3.0  | 0.02                |
| Sika . . .  | 0.5   | »  | 1590                    | 1530         | —   | 2926           | 3.0  | 0.0                 |
| Pontural .  | 52.7  | Porosité                                 | 1300                    | 1700         | 2230  | 2116           | 10.5   | 0.0                 |
| Dentacryl.  | 0.7   | Pas de changement                        | —                       | 480          | 451   | —              | 7.0  | 6.0                 |

L'étude d'ensemble montre que tous les alliages examinés ou bien ont changé de couleur ou bien sont devenus poreux quand on les soumettait à la corrosion en solution acide. Sur la foi de cette épreuve il faut considérer le Pontural, avec sa forte déformation sous l'effet de la pression, comme le plus mauvais des alliages soumis à l'examen. De trois autres, le Randolf possède à la fois la plus grande dureté et la résistance la plus élevée à la tension; en outre sa résistance à la pression et ses qualités de coulée sont satisfaisantes. Contrastant avec ces avantages il y a sa forte corrosion.

Parmi les quatre alliages le Silca doit être estimé le plus avantageux pour sa dureté, sa résistance à la compression, sa coulée lente et sa grande résistance à la corrosion. Sa résistance à la

tension est quelque peu inférieure à celle du Randolf, mais il est douteux que cette différence ait la moindre importance.

Le Dentacryl offre une résistance à la tension et à la flexion fortement inférieure à celle des alliages examinés; sa facilité de coulée est considérable. En contre-partie, il paraît se défendre très bien contre la corrosion.

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