

1952 Geo. C. Paffenbarger

NATIONAL BUREAU OF STANDARDS REPORT

2286

REPORT ON DENTAL RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

Dental Research Laboratory

**For the Half Year Ending
December 31, 1952**



**U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS**

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THE NATIONAL BUREAU OF STANDARDS

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Mineral Products. Porcelain and Pottery. Glass. Refractories. Enameled Metals. Concreting Materials. Constitution and Microstructure. Chemistry of Mineral Products.

Building Technology. Structural Engineering. Fire Protection. Heating and Air Conditioning. Floor, Roof, and Wall Coverings. Codes and Specifications.

Applied Mathematics. Numerical Analysis. Computation. Statistical Engineering. Machine Development.

Electronics. Engineering Electronics. Electron Tubes. Electronic Computers. Electronic Instrumentation.

Radio Propagation. Upper Atmosphere Research. Ionospheric Research. Regular Propagation Services. Frequency Utilization Research. Tropospheric Propagation Research. High Frequency Standards. Microwave Standards.

Ordnance Development. These three divisions are engaged in a broad program of research and development in advanced ordnance. Activities include
Electromechanical Ordnance. basic and applied research, engineering, pilot production, field testing, and evaluation of a wide variety of ordnance matériel. Special skills and facilities of other NBS divisions also contribute to this program. The activity is sponsored by the Department of Defense.
Ordnance Electronics.

Missile Development. Missile research and development: engineering, dynamics, intelligence, instrumentation, evaluation. Combustion in jet engines. These activities are sponsored by the Department of Defense.

● Office of Basic Instrumentation

● Office of Weights and Measures.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

NBS REPORT

0708-10-0707
0708-10-0724
0708-10-3822
0708-10-3824

February 15, 1953

2286

REPORT ON DENTAL RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

Dental Research Laboratory

For the Half Year Ending
December 31, 1952

The dental research program at the National Bureau of Standards is carried on in cooperation with the Council on Dental Research of the American Dental Association, the Army Dental Corps, the Air Force Dental Service, the Navy Dental Corps, and the Veterans Administration.



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REPORT ON DENTAL RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

1. INTRODUCTION

The activities of the Dental Research Laboratory at the National Bureau of Standards for the six-month period ending December 31, 1952 are covered in this report. As various phases of the work were completed, detailed reports have been issued to the agencies cooperating in the program. Copies of reports on work completed during this period are attached. In addition, the results obtained on work now in progress are briefly summarized.

2. REPORTS ISSUED

- NBS No. 2032 The Color Stability of Direct Filling Resins
- NBS No. 2089 Thermal Expansion of Silica-Gypsum Investments: Effect of a New Factor, Preheating
- NBS No. 2242 Mechanism of Hygroscopic Expansion in Dental Casting Investments
- NBS No. 2283 Temperatures Developed in Rotating Dental Cutting Instruments

3. WORK IN PROGRESS

3.1 Structure of Human Tooth Enamel and Dentin

Investigation of the fundamental structural characteristics of human tooth enamel and dentin.

- (a) Fluorescence Investigation. The spectra of decalcified specimens of enamel and dentin were examined to obtain information on the source of fluorescence of tooth structures. Although the fluorescence spectra of these specimens of enamel protein and dentin protein were found to be very similar to those of undecalcified enamel and dentin, the energy maximum, in each case, was displaced towards the blue end of the spectrum by approximately 500 reciprocal centimeters. This suggests that the observed fluorescence spectrum of normal teeth is the weighted average of the protein (organic) and the apatite (inorganic) constituents of the teeth. To test this hypothesis samples of enamel and dentin free from organic material are now being prepared.

Observations on tooth sections from teeth frozen immediately after extraction showed that carious and "pre-carious" lesions frequently fluoresced a bright red at their edges. The intensity of the red fluorescence decreased rapidly if the tooth section was kept at room temperature but could be preserved for several days in frozen specimens. Even at this low temperature, however, the red fluorescence almost disappeared after one or two weeks.

A number of strains of bacteria known to be associated with tooth decay were examined, but none exhibited fluorescence similar to that observed in the carious areas. Attempts to culture the red fluorescing material at the National Institutes of Health have so far been unsuccessful.

- (b) Crystallographic Investigation. The study of particle size distribution in teeth, bone and apatite minerals was continued. The method of low angle scattering of x-rays was employed. All carbonate-containing basic calcium phosphates (bone, teeth, etc.) examined seem to have a particle size distribution which extends almost into the molecular range, i.e. 50 angstroms.

A new method being pursued makes possible the determination of particle size distribution from 10,000 Å to 10 Å through a detailed study of the broadening of the Bragg (high angle) x-ray reflections of the material. The method involves the study of the corrected shape of the Bragg reflection which is represented by a cosine Fourier series. The set of Fourier coefficients obtained will then give information as to the spread of particle size in the range mentioned.

By coupling the results of the above x-ray methods with other approaches, such as low temperature nitrogen adsorption and preferential solubility studies, it is hoped that a better understanding may be had of the placement of the trace elements in teeth, bone and allied apatites.

3.2 Surface Crazing of Acrylic Resins

Determination of the mechanism involved in the surface crazing of acrylic resins.

Water sorption measurements of various methyl methacrylate polymers were continued. Sorption as well as craze susceptibility of commercial samples is dependent on the presence and concentration of copolymer and plasticizers. It was established that remolding of specimens at 150°C increases markedly the water sorption at 100°C. Polymer disks of varying molecular weight showed little difference in sorption values on storage at 4°C. Measurements at other temperatures are now being conducted. For plastic teeth no correlation could be found

between water sorption at 100°C and craze resistance. All samples investigated gave a continuous weight increase even after storage for 37 days. In general the rate of sorption decreased with time. A few samples, however, showed a considerable increase in the sorption rate after a definite time interval. Specimens which had been heated at 100°C in water for over 200 days and then dried to constant weight and stored in water at 37°C showed increased water sorption with a decrease in molecular weight.

The internal strain in resins is being studied by imbedding SR-4 resistance strain gages in samples of methyl methacrylate. The change in strain caused by the sorption of water at various temperatures will be used to determine why the resin, when cured in contact with a water-containing surface, shows poor craze resistance.

Data on the water vapor sorption of specimens of varying molecular weight were obtained to determine the effect of relative humidity and to correlate the results with those obtained for disks immersed in water. Below the transition temperature vapor sorption appears to be independent of molecular weight below a relative humidity of 80 percent. Water uptake is nearly linear from 10 to 50 percent relative humidity. Above 70 percent relative humidity there is a rapid increase in the amount of water sorbed.

3.3 Polymerization of Acrylic Resins

Investigation of the chemical reactions which are responsible for the induction period of the polymerization of methyl methacrylate.

The hydrolysis of polymethyl methacrylate specimens exposed to boiling water for 40 days was investigated. Reaction products identified were methyl alcohol, methacrylic acid and traces of carbonyl compounds, possibly pyruvic acid or formaldehyde.

Residual catalyst was found in all commercial polymer samples analyzed. This explains the lower degradation temperature of peroxide catalyzed polymer.

The usefulness of the free radical α, α -diphenyl β picryl hydrazyl (D.P.P.H.) in the study of radical formation and reaction was investigated. Preliminary results obtained with 2:2-azo-bis (isobutyronitrile) (A.I.B.N.) in benzene solutions of D.P.P.H. indicate that half-time decomposition of D.P.P.H. may offer a new and convenient approach to measure rate of free radical formation, reactivity and efficiency of inhibitors.

3.4 Physical, Chemical and Engineering Properties of Dental Materials

Determination of the properties and processing characteristics and development of more satisfactory technics for the use of materials employed in the practice of restorative dentistry.

- (a) Metallurgical Investigations. The investigations of amalgams for dental use have been extended to include a study of the possibility of using gallium as a substitute for mercury. Gallium has the advantages of being nontoxic and of wetting tooth structure. Experimental alloys which set at room temperature have been made of gallium. Alloys of copper-tin, nickel, nickel-silicon, and silver-tin, with gallium are being investigated to determine whether properties suitable for dental restorations can be obtained. An alloy which can give better adaptation to the cavity walls of the tooth is greatly needed.
- (b) Chrome Alloys for Dental Castings. Chrome alloys from a number of commercial sources are being studied for determination of such mechanical properties as modulus of elasticity, strength, elongation and hardness. This project will be extended to include a wider group of alloys and accessory materials to provide the Federal dental services with sufficient data to specify a simple and satisfactory material for clinical use.
- (c) Revolving Dental Cutting Instruments. The work on revolving cutting instruments has been progressing very satisfactorily, using the standardized procedure for evaluating dental burs previously developed. Study of the diamond abrasive instruments will be completed shortly. Diamond instruments of various manufacturers appear to vary widely in their effectiveness in cutting pyrex glass. Instrument wear is accelerated when eccentricity is excessive, and vibrations amplitude rises as eccentricity increases.
- (d) Abrasion or Wear Resistance of Plastic Teeth. An accelerated test was developed to show differences in resistance to abrasion of various brands of plastic teeth. The teeth previously soaked in water to equilibrium are placed in a ball mill with 1/4-inch steel balls and number 200 Aloxite powder and tumbled for 2 hours.
- (e) Development of Ultrasonic Test Methods. Ultrasonic technics for evaluating the elastic constants of polymethyl methacrylate are being studied. The construction of an experimental instrument which employs the diffraction of monochromatic light by ultrasonic waves has been completed.

This method is an outgrowth of the theory of Fues and Ludoff. A nondestructive test procedure would be very useful in following the change in properties produced by processing variables in this and other resins.

- (f) Clinical Research on Self-Curing Denture Base Resins. The clinical study of self-curing denture base resins in cooperation with the Veterans Administration has been progressing satisfactorily. More than 100 practical dentures are being observed and measured for dimensional changes periodically. Observation of 30 dentures made at the Bureau and 91 made in VA hospitals indicates a very high percentage of satisfactory service.
- (g) Periapical X-ray Film Survey. A preliminary survey of periapical x-ray film has been initiated to determine the practitioners' requirements. Properties under investigation include emulsion speeds, light and water resistance of the film wrapping, and the shelf life. Common x-ray practice procedures are to be determined through a questionnaire which will be sent to general practitioners and specialists. If warranted, a specification will be developed.
- (h) Hydraulic Turbine Contra-Angle Handpiece. A hydraulic handpiece which develops sufficient torque at speeds above 14,500 r.p.m. to cut dental enamel with diamond instruments has been designed. Two experimental contra-angle handpieces have been constructed of different turbine designs and with several types of bearings. The driving mechanism is a closed system; water flows to the turbine in the handpiece through nylon tubing from a hydraulic pump driven by 1/2 H.P. motor. This provides a power system relatively free of vibration for revolving cutting instruments.
- (i) Clinical Camera. A 35 mm clinical camera, which is uniquely suitable for the clinical testing program, has been completed. Magnifications up to four diameters with adequate depth of focus can be obtained. The photo-flash bulbs used as a light source simplify the exposure factors. Focusing is done through the lens using a reflex-type camera.
- (j) Evaluation of Materials. Materials evaluated for the cooperating agencies by specification or special test methods included plastic teeth, porcelain teeth, direct filling resins, silicate cement, amalgam alloys, mercuries, cavity liners, elastic impression materials, impression pastes, impression compounds, duplicating material, denture base resin, base plate waxes, inlay waxes, wax solvents, gold alloys, chromium-cobalt alloys and pickling salts.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

NBS REPORT

0708-20-3824

2032

Progress Report

on

THE COLOR STABILITY OF DIRECT FILLING RESINS

by

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This work is a part of the dental research program conducted at the National Bureau of Standards in cooperation with the Council on Dental Research of the American Dental Association, the Army Dental Corps, the Air Force Dental Corps, the Navy Dental Corps and the Veterans Administration.

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The Color Stability of Direct Filling Resins

Abstract

Data are presented showing the amount of color change occurring in direct filling resins during storage at room temperature in the dark and when exposed to the radiation of a sunlamp. These data were collected with a Hunter Multipurpose Reflectometer. Most of the changes taking place during storage occurred within one day after formation of the specimens and amounted to approximately 2 NBS units of color difference. A color change of about 5 NBS units resulted from exposure to the sunlamp. The value of the instrument for work on dental materials was greatly increased by the addition of a special adapter which makes possible the determination of color and color changes of small specimens or of small areas of specimens.

1. Introduction

The test commonly employed for determining the color stability of resins used in dentistry is based upon a visual comparison of the color of a specimen exposed to the radiation of a sunlamp and the color of an unexposed sample [1]¹. This is not a satisfactory test with the newer acrylic resins which polymerize at room temperature as a result of the addition of accelerators and of a higher catalyst concentration, because of the instability of these materials and their tendency to form colored decomposition products at room temperature (Taylor and Frank [2]). These chemical reactions, which produce discolorations after polymerization, occur slowly on unexposed specimens even though they are carefully protected and stored in the dark. This may cause the standard or unexposed specimen to change during the period required for the exposure test. The use of permanent color standards is not feasible because of the varied color shades used by the manufacturers and because of the mottled appearance used for good reproduction of the oral tissues.

The visual comparison test is wholly unsatisfactory for determining the amount of color change, because no quantitative results are obtained. To overcome these difficulties of measuring color stability the Hunter Multipurpose Reflectometer, which supplies a numerical value for color, was employed. This paper describes the method and results obtained with direct filling resins.

The use of an instrument which measures color numerically will be very helpful in solving many of the problems in dentistry where color is an important factor. For example, accurate color determinations would make it possible to prepare a single shade guide for silicate and direct filling resins and artificial teeth instead of the many different shade guides used by the various manufacturers. Measurement of mouth tissue colors can be expected to result in the production of more natural colors for artificial dentures.

2. Procedure

The Hunter Multipurpose Reflectometer [3] was used in this investigation. Because of the necessity of preparing small specimens in order to approximate the polymerization conditions occurring in a filling in the mouth, an adapter was developed to be used in conjunction with the condensing lens which may be used with the reflectometer.

Specimens (39 x 12 x 1-1/2 mm) were prepared from each brand of material using a tin-foil lined brass mold. Mixing of the material was done according to the instructions of the manufacturer. Color measurements were made one hour after each specimen was prepared and at intervals during seven months of storage in air in the dark at room temperature. At the end of this time the specimens were subjected to the color stability test of American Dental Association Specification No. 12 for Denture Base Resin, which consists of exposing the specimens to the radiation of an S-1 bulb in a General Electric sunlamp at a distance of seven inches for 24 hours. Color measurements were made immediately before and after this test. These specimens were again stored in air at room temperature in the dark and color measurements were made about 1-1/2 months later.

3. Results

The materials listed in Table 1 were used in this study.

The results of the color measurements are shown in Figures 1, 2, 3 and 4. In all cases the first measurement was made when the specimen was one hour old and this measurement was used as the standard to which all color comparisons were made. Sevricon and Swedon color comparisons were made on exposed and unexposed portions of specimens subjected to the American Dental Association color stability test. Sevricon changed in color 4 NBS units while Swedon changed 8 NBS units.

Figures 1 and 3 show average values of color changes and yellowness index change. Values for Athermoplast were omitted from these averages because of the extremely large color change in this resin during storage and because the material is no longer on the market.

Figures 2 and 4 show the cumulative effect of each treatment on the various brands of material.

4. Discussion

The magnitude of any color difference between two colors may be found in NBS units by calculation from the CIE tristimulus values or from colorimetric comparison of the two colors (see [4] and [5]). Color and color difference determinations may be made with the Hunter Multipurpose Reflectometer, and may be determined to approximately 1 NBS unit by the use of this instrument. A trained observer is able to see with difficulty a color difference of 1 NBS unit. Less than about 10 NBS units is usually seen as a variation of the same color, while more than 10 NBS units of color difference is usually seen as change to another color.

The plots of color difference (Figures 1 and 2) show that the specimens changed in color about 2 NBS units of color difference during storage at room temperature in the dark. Most of this change occurred during the first 24 hours (Figure 1).

When the specimens were exposed to the American Dental Association color stability test, which is designed to accelerate the color changes that would occur on exposure of the resin to light during normal use, the average change in color was approximately 5 NBS units.

It should be noted (Figure 1) that the color change during the period of storage at room temperature in the dark after the exposure test amounted to 1 to 3 NBS units for all of the materials with the exception of Athermoplast V10. It thus appears that the reactions initiated by exposure of the resin specimens to the radiation of the test lamp may continue for some time after the completion of the exposure test.

Lightness index changes (variation in gray) which were very small occurred in six of the brands of resin. The lightness index has been defined as the square root of the reflectance. The seventh brand, Athermoplast V10, darkened materially.

The changes in the yellowness index (Figures 3 and 4) show that the specimens become yellower. The similarity of Figures 1 and 3 and of Figures 2 and 4 indicates that the color changes are, to a large extent, changes in yellowness and analysis of the original data shows this to be true.

This investigation deals only with the color changes occurring during storage of the direct filling resins in air at room temperature in the dark and by exposure to light. Other types of discoloration caused by the staining of foods and other materials in the mouth are not considered in this report.

5. Summary

By the use of the Hunter Multipurpose Reflectometer the color and the color changes occurring in direct filling resins have been quantitatively determined.

Data have been presented showing that the direct filling resins change color with age in the absence of light. The average value of color difference is about 2 NBS units. Most of the color change occurs during the first 24 hours.

The effect of the American Dental Association color stability test (exposure to the radiation of a sunlamp) on the several brands of material tested varies from approximately 1 to 11 NBS units of color difference. The average value of about 5 NBS units is an easily noticeable color change.

References

1. Research Commission, Revised American Dental Association Specification No. 12 for Denture Base Material, Acrylic Resin or Mixture of Acrylic and Other Resins (effective Jan. 1, 1942), J. A. D. A., 29:127, (Jan) 1942.
2. Taylor, P. B., and Frank, S. L., Low temperature polymerization of acrylic resins, J. Dent. Research, 29:486-492, (Aug.) 1950.
3. Hunter, Richard S., A Multipurpose Photoelectric Reflectometer, J. Research NBS, 25:596 (Nov.) 1940.
4. Judd, Deane B., Colorimetry, National Bureau of Standards Circular 478, page 29, issued March 1, 1950. (For sale by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.) (Price 30 cents)
5. Colors of Polystyrene Plastics, Commercial Standards, CS 156-49 (For sale by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.) (Price 5 cents)

Table 1
Direct Filling Resins

<u>Brand</u>	<u>Manufacturer</u>
Athermoplast V10	Athermoplast Products Inc.
Dentafil	Dental Fillings, Ltd.
Duz All	Coralite Dental Products Company
Fastcrown	Acrylite Company, Inc.
Kadon	The L. D. Caulk Company
Plastofilling pf3	Plastodont, Inc.
Replica	Cosmos Dental Products, Inc.
Sevriton*	Amalgamated Dental Co., Ltd.
Swedon*	Svedia Dental Industri AB.

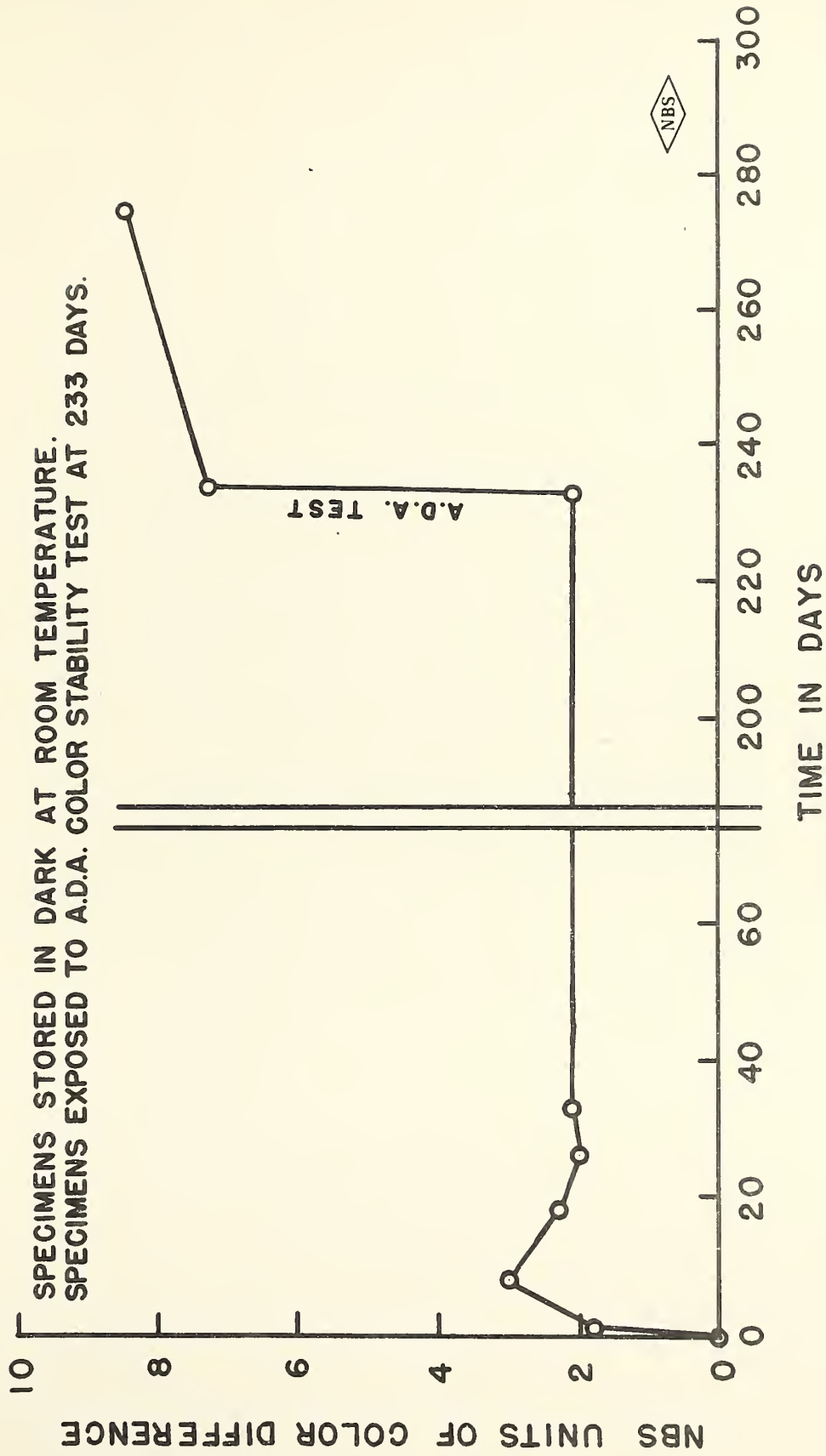
* Color comparison were made only on exposed and unexposed portions of specimens after American Dental Association color stability test.

Figure 1

Average difference in color stability of the direct filling resins.

COLOR STABILITY

DIRECT RESINOUS FILLING MATERIALS



$\frac{1}{2} \times 10 = 5$

[illegible]

Figure 2

Difference in color stability of the direct filling resins.

COLOR STABILITY

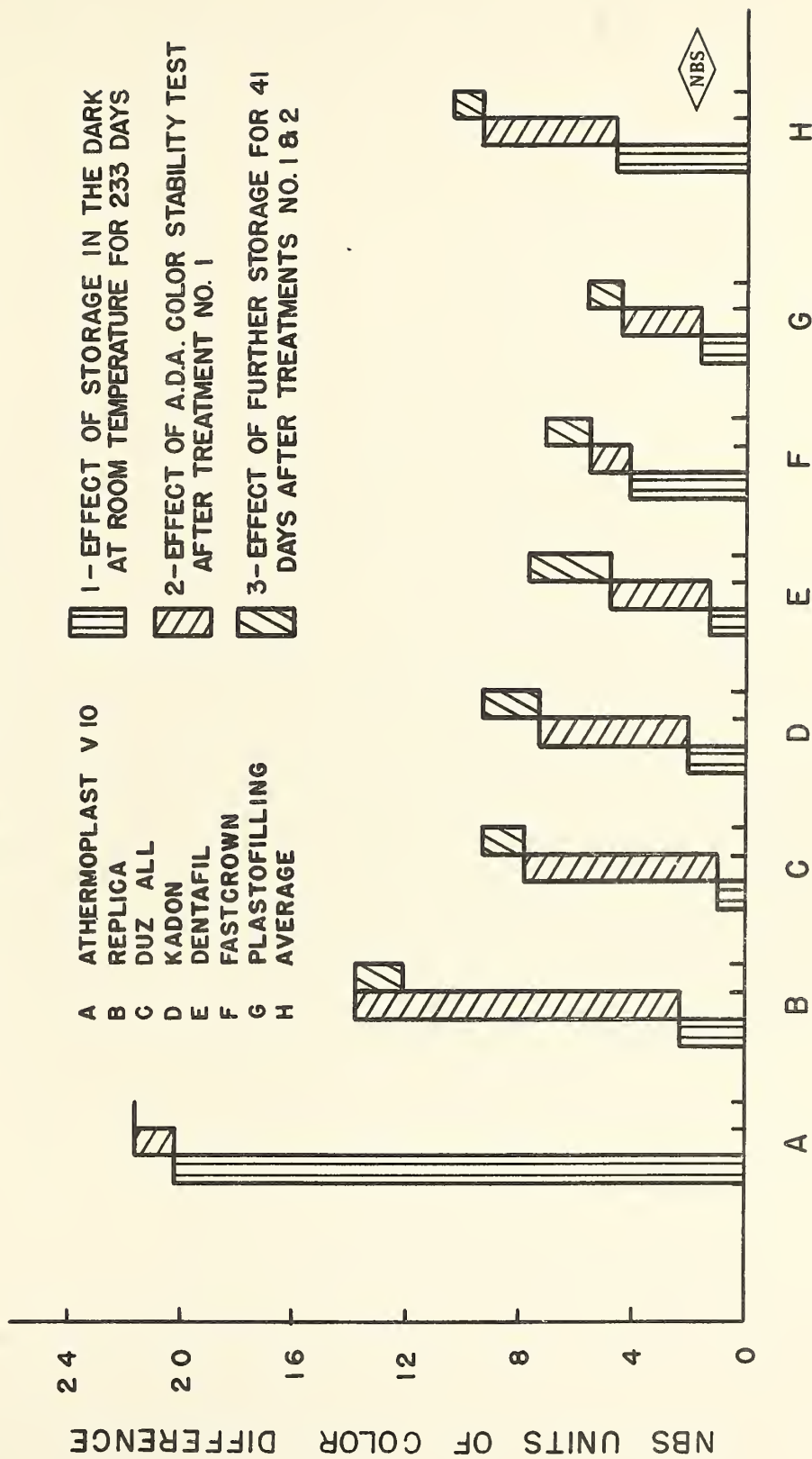


Figure 3

Average yellowness index of the direct filling resins.

YELLOWNESS INDEX DIRECT RESINOUS FILLING MATERIALS

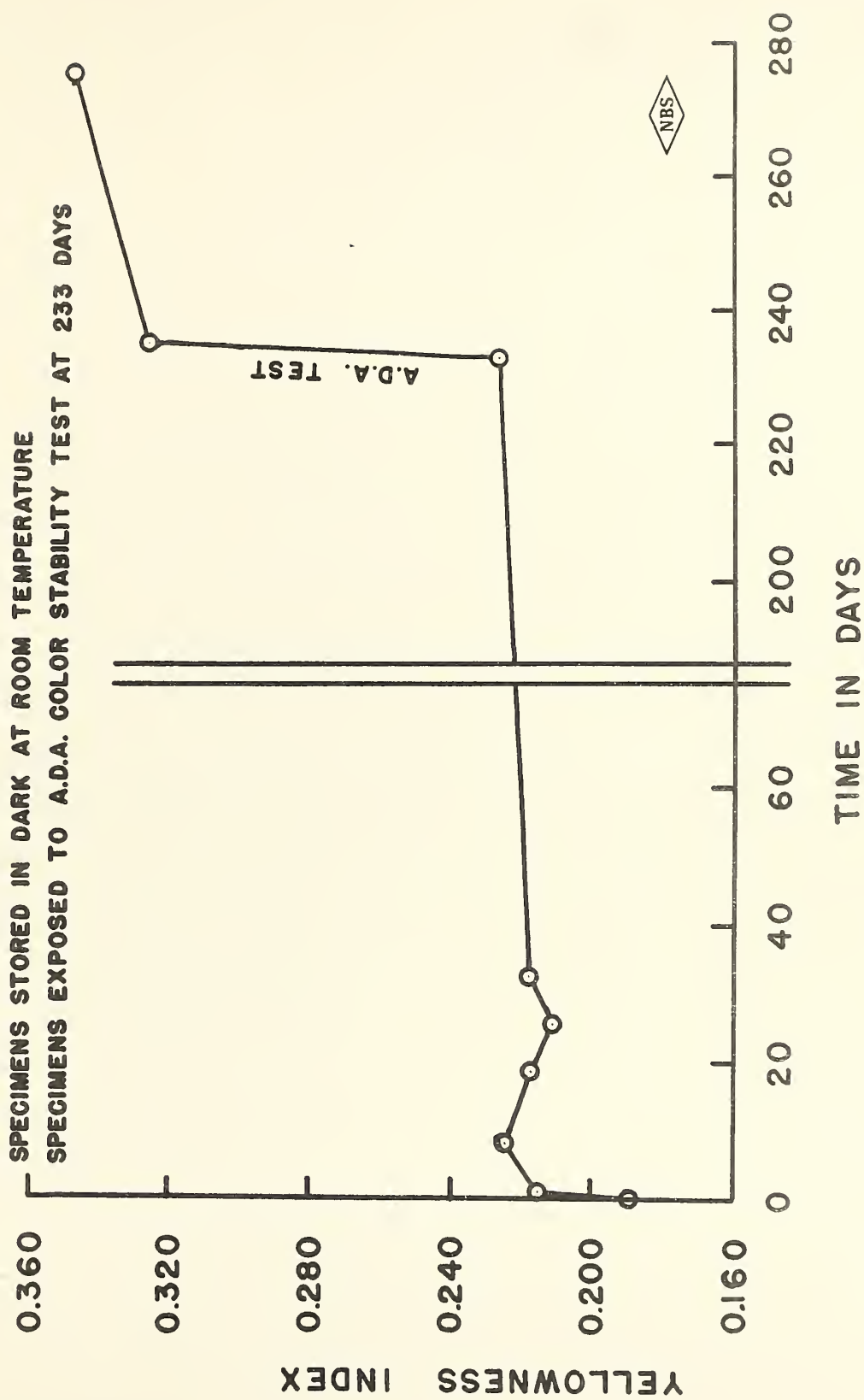
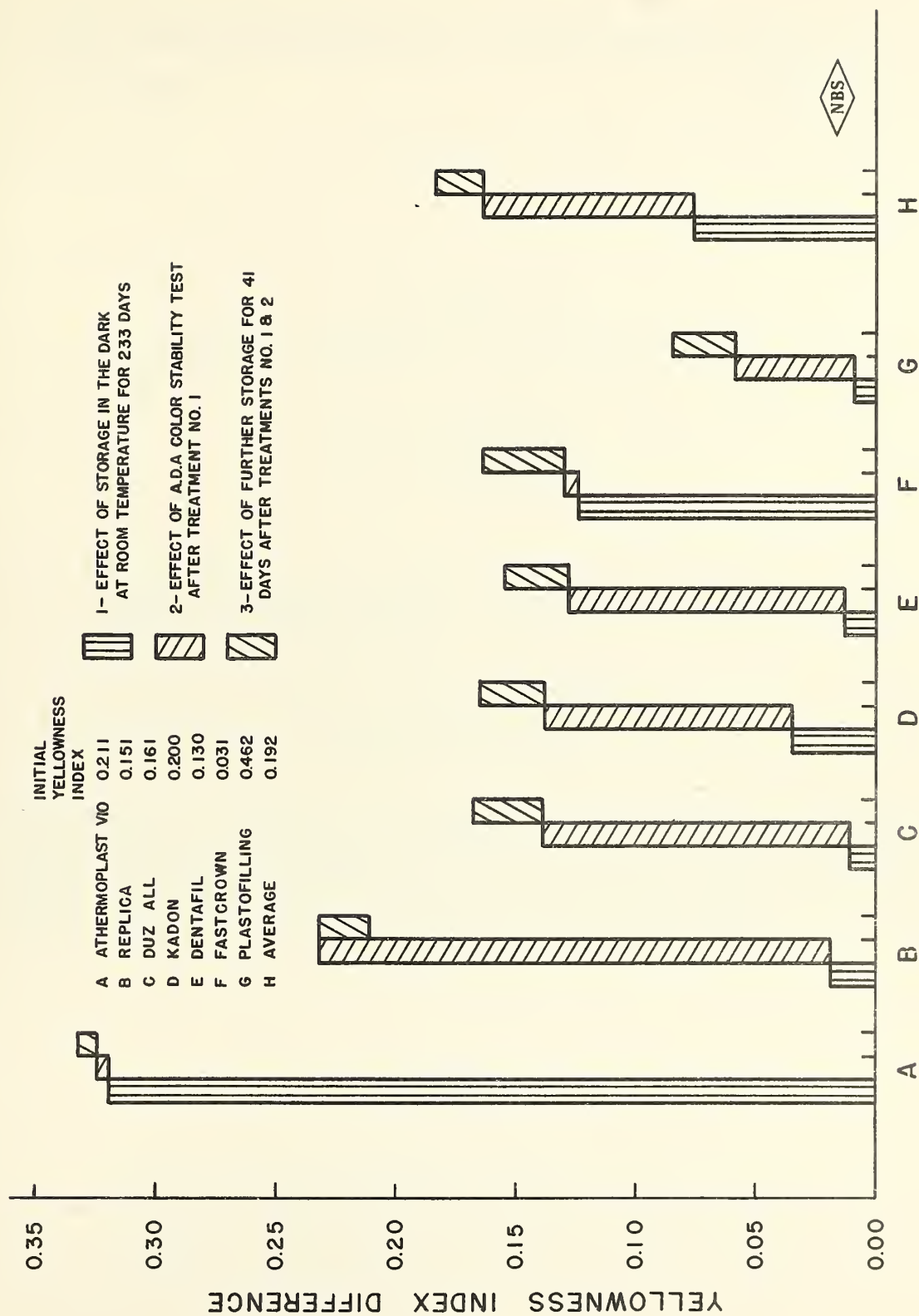


Figure 4

Yellowness index changes of the direct filling resins.

CHANGES IN YELLOWNESS INDEX



NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

NBS REPORT

0708-10-3824

December 2, 1952

2089

PROGRESS REPORT
ON
THERMAL EXPANSION OF SILICA-GYPSUM
INVESTMENTS: EFFECT OF A NEW FACTOR, PREHEATING

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This work is a part of the dental research program conducted at the National Bureau of Standards in cooperation with the Council on Dental Research of the American Dental Association, the Army Dental Corps, the Air Force Dental Corps, the Navy Dental Corps and the Veterans Administration.



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Abstract

It has been observed that the thermal expansion properties of some silica-gypsum investments are changed by a pre-heat treatment (three hours at 120° C) which apparently causes a partial loss of water of crystallization of the gypsum. The presence of boric acid and sodium chloride seems to be associated with the increased thermal expansion produced in some investments and decreased thermal expansion produced in other investments. X-ray diffraction and petrographic analysis gave no evidence of the reason for this change. The altered thermal expansion affects both the dimensional accuracy and the formation of fins on castings.

1. Introduction

Ever since the adoption of an inlay casting investment specification by the American Dental Association in 1930, the thermal expansion compensation technique has enjoyed widespread usage in the casting of dental restorations.

The addition of boric acid by Weinstein in 1929 [1] and sodium chloride by Moore in 1933 [2] to quartz-plaster investments, and the development of an investment containing cristobalite in 1933 [3, 4] have done away with investments that shrink when they are heated. Various techniques for precision dental casting have been introduced from time to time, but none have displaced the thermal expansion compensation technique from being the most used. Yet, despite its favor and usage over a period of twenty-odd years, and despite the fact that it has been investigated for a still longer period of time, the technique contains some undiscovered variables. The purpose of this investigation has been to obtain information on one such variable in the alteration of the thermal expansion of plaster-silica investments; namely, the effect produced by drying at elevated temperatures and then cooling prior to the normal continuous burnout procedure. In some investments the subsequent thermal expansion is increased, and in other

investments the thermal expansion is decreased. Such changes can critically affect the results in the field of precision casting, and it is therefore important to recognize and evaluate these changes.

The changes in thermal expansion may result from dehydration of the calcium sulphate. It has been demonstrated by Weiser et al. [5] that the dehydration of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ follows definite steps; above 100°C the CaSO_4 is in the hemihydrate form.

2. Procedure

The following investments were used in this study:

<u>Material</u>	<u>Manufacturer</u>
Cristobalite Model Investment	Kerr Manufacturing Company
Pyromold No. 2 Investment	Wm. Getz Corporation
R & R Gray Investment	Ransom & Randolph Company

All investment specimens and molds used in the investigation were mixed according to the manufacturer's recommendation for water-powder ratio, using room-temperature tap water, and were mechanically spatulated and vibrated.

The thermal expansions of the investments and the changes in length resulting from the preheating and cooling cycle were determined using the fused quartz tube apparatus as described by Hidnert and Sweeney [6]. The temperature of the specimen was measured with an iron-constantan thermocouple, the junction being placed in the center of the specimen. Specimens were heated at a rate of approximately 10°C per minute.

Setting expansions of the three investments were determined by the comparator microscope method described by Coleman [7].

In addition to the investigation of commercial investments, thermal expansion data were obtained on plaster and hydrocal as received, and with the addition of two common constituents of dental investments, sodium chloride and boric acid. These were studied by x-ray diffraction and petrographic methods before and after heating.

The partial denture and M.O.D. inlay castings were made by conventional dental procedures using various combinations of the investments studied. The partial denture castings were examined for fin formation. The dimensional accuracy was evaluated by placing the castings on M.O.D. steel dies.

3. Results and Discussion

Thermal Expansion

The dimensional changes caused by heating are represented graphically in Figures 1, 2 and 3 (curves marked: As Cast, 1). In all cases the values are the average of several tests. By drying the specimens at 120° C for three hours and then cooling them to room temperature before heating in the thermal expansion apparatus, different amounts of thermal expansion resulted (curves marked: Preheated, 2). This treatment simulates one used by prosthetic laboratories for drying investment casts prior to waxing up a partial denture framework pattern.

It is also accepted procedure by some prosthetic laboratories to dip investment casts into a mixture of rosin and wax at 150-165° C after drying, to facilitate the adhesion of patterns to the cast. A series of specimens treated in a like manner exhibited thermal expansions as represented in curves marked: Rosin-wax dipped, 3, in Figures 1, 2 and 3. It will be noted that in Figures 2 and 3 the expansion curves of both the preheated and rosin-wax dipped specimens are relatively the same, making it evident that the change in thermal expansion properties is a result of the preheating.

The fact that the thermal expansion of the rosin-wax dipped specimen in Figure 1 is unlike that of the preheated specimen cannot be explained at this time.

Change in Length as a Result of Preheating

It has been suggested that the change in thermal expansion of these investments is due to the loss of water during the preheat treatment. Sweeney and Taylor [8] have shown that the loss of water of crystallization of gypsum products is accompanied by a loss in linear dimension. Specimens of the three investments heated at 120° C for three hours and cooled to room temperature exhibited the following changes in length:

<u>Material</u>	<u>Change in Length</u> %
Cristobalite Model Investment	±0.00
Pyromold No. 2 Investment	-0.04
R & R Gray Investment	-0.10

These are the average values of four specimens on which the measurements are accurate to 0.02 percent. There is no apparent correlation between the dimensional change caused by preheating and the differential thermal expansion.

Setting Expansion

Setting expansions of the three investments determined by the comparator microscope method described by Coleman [7] were as follows:

<u>Material</u>	<u>Setting Expansion</u> %
Cristobalite Model Investment	0.21
Pyromold No. 2 Investment	0.26
R & R Gray Investment	0.21

Whether or not the full setting expansion is effective in partially compensating for casting shrinkage of gold is controversial. However, with these materials this will not appreciably affect the total expansion obtained because all the setting expansion values are very nearly the same.

Effects of Composition

A chemical analysis, Table 1, shows the amounts of boric acid and sodium chloride used in these investments. Inasmuch as silica is a very inert material, it is thought that the boric acid or sodium chloride reacts with the calcium sulphate in these investments. Therefore, the effect of these materials on the thermal expansion of plaster and hydrocal was studied. Approximately the same ratio of boric acid or sodium chloride to gypsum present in the investments was added to the plaster and hydrocal. Figure 4 shows the thermal expansions of French's plaster and R & R Castone as received, with the addition of 1.5 percent sodium chloride, and with the addition of 5.2 percent boric acid. In plotting the curves the fiducial reading of the expansion was taken as the original length of the specimen. The loss in length in the preheated specimens is indicated by these fiducial readings of the respective curves.

The plaster had less thermal expansion than hydrocal when given the same treatment. For example, curve 1-a is less than 2a; curve 3b is less than 4b; etc.

The expansions of both plaster and hydrocal were increased very slightly by the addition of 1.5 percent sodium chloride. However, the preheating did not affect these expansion curves significantly, although a shrinkage did occur during the preheating cycle.

The effect of adding 5.2 percent boric acid on the thermal expansions of plaster and hydrocal is quite evident. The loss in length of the specimens due to preheating is less and the specimens withstood a much higher temperature without deterioration. The expansion of plaster and hydrocal is generally greater at all temperatures measured (Figure 4).

The most significant effect of the addition of boric acid was to increase the temperature to which the specimens that had been preheated could be heated before loss of strength occurred. This is in agreement with the work of Coleman [7] who noted that the addition of boric acid to silica-gypsum investments caused an increase in strength at elevated temperatures.

In an attempt to correlate the expansion data obtained with changes in crystallographic structure, the specimens described in Figure 4 were subjected to x-ray diffraction and petrographic analysis. Probably because of the relative impurity of the samples, only minor differences could be detected, and these were not significant.

Experimental Castings

In order to determine the immediate practical significance of these changes in thermal expansion properties produced by preheating, technic partial denture castings were made. A basic set of eighteen castings were made using the three investments. Six casts of each investment were poured, three of which were dried at 120° C for three hours and dipped in a rosin-wax mixture, consisting of 75 percent commercial lump rosin and 25 percent paraffin, at 155° C for thirty seconds. After waxing up the patterns, one cast without treatment was invested in each of the three investments, and one cast with the drying and dipping treatment was invested in each of the three investments. In this manner, different combinations of mold and cast thermal expansions were compared. From these and many additional technic castings it was shown that if the thermal expansion of the cast exceeds the thermal expansion of the mold, even by as little as 0.2 percent, the mold will crack, allowing a fin to form on the casting. Conversely, and contrary to a common theory on the cause of fins, if the thermal expansion of the mold exceeds that of the cast by as much as 0.5 percent, no fins will be formed. It is therefore important to maintain like thermal expansions of the cast and the mold in casting partial denture frameworks to prevent casting failures due to fin formation. Figure 5 shows a cross-sectioned mold and partial denture cast that has been heated to casting temperature, where the thermal expansion of the cast exceeds that of the mold. The cracking of the mold is evident at the corner of the cast. Figure 6 shows two technic castings made from identical wax patterns. The casting on the right is satisfactory; the casting on the left exhibits the typical fin formation resulting from a high-expanding cast and low-expanding mold.

Castings made on M.O.D. steel dies demonstrate that the dimensional accuracy of a casting can be affected by preheating alone. Figure 7 illustrates three M.O.D. inlays in

place on steel dies. The molds in this case were given the regular burnout procedure. It can be seen that castings C (Cristobalite Model Investment) and R (R & R Gray Investment) fit the dies well. The thermal expansion of these investments is 1 percent at 700° C. Casting P (Pyromold No. 2 Investment) does not fit the mold. The thermal expansion of this investment is 0.7 percent at 700° C. Figure 8 shows three M.O.D. inlays in place on the same steel dies, the molds of which were subjected to a 120° C preheat treatment followed by cooling to room temperature prior to the regular burnout procedure. The fit of casting C remains unchanged; that of casting P is improved, and that of casting R is worse, as can be predicted from the differential thermal expansions shown in Figure 9. From these experiments it must be pointed out that the burnout procedure should be a continuous one to prevent changes in the thermal expansion properties of the mold.

4. Summary

1. It has been observed that the thermal expansion of some casting investments is markedly changed by a preheat treatment consisting of heating the specimens of investment for three hours at 120° C and then cooling them to room temperature.

2. Of the three investments studied, one showed an increase in the thermal expansion, another practically no change, and the third, a decrease in thermal expansion.

3. The actual loss in dimension caused by the preheating of investments cannot be correlated with resulting changes in thermal expansion.

4. The thermal expansion changes due to preheating have been demonstrated to be one cause of fin formation on partial denture castings.

5. X-ray diffraction patterns and petrographic analysis fail to show appreciable changes in gypsum crystals due to preheating.

6. It is suggested that the difference in thermal expansion may be caused by unknown crystalline phases of the gypsum, or by mineralization of the calcium sulphate crystals by impurities or minor constituents.

5. Bibliography

1. U. S. Patent No. 1,708,436.
2. U. S. Patent No. 1,924,874.
3. U. S. Patent No. 1,932,202.
4. Sweeney, W. T., Cristobalite for Dental Investment, J. Am. Dent. Assn. 20, 108 (1933).
5. Weiser, Harry B.; Milligan, W. O.; and Ekholm, W. C., Mechanism of the Dehydration of Calcium Sulphate Hemihydrate, J. Am. Chem. Soc. 58, 1261 (1936).
6. Hidnert, Peter; and Sweeney, W. T., Thermal Expansion of Magnesium and Some of its Alloys, NBS J. Res. 1, 771 (Nov. 1928).
7. Coleman, R. L., Physical Properties of Dental Materials, NBS J. Res. 1, 867 (Dec. 1928).
8. Sweeney, W. T.; and Taylor, D. F., Dimensional Changes in Dental Stone and Plaster, J. Dent. Res. 29, 749 (1950).

Table 1

Chemical Analysis

Investment Brand	Insoluble Siliceous Matter	R ₂ O ₃ *	Ca	SO ₄	Mg	Combined H ₂ O	H ₃ BO ₃	NaCl
	%	%	%	%	%	%	%	%
Cristobalite Model	61.0	trace	10.6	25.5	0.2	2.4	0.02	0.02
Pyromold No. 2	58.3	0.5	10.7	25.7	0.3	2.6	2.0	Not detected
R & R Gray	54.6	trace	12.3	29.7	0.2	2.7	Not detected	0.62

*Oxides of elements which may be precipitated by ammonium hydroxide, such as iron, aluminum and titanium.

Figure 1

Thermal expansion of dental investment.

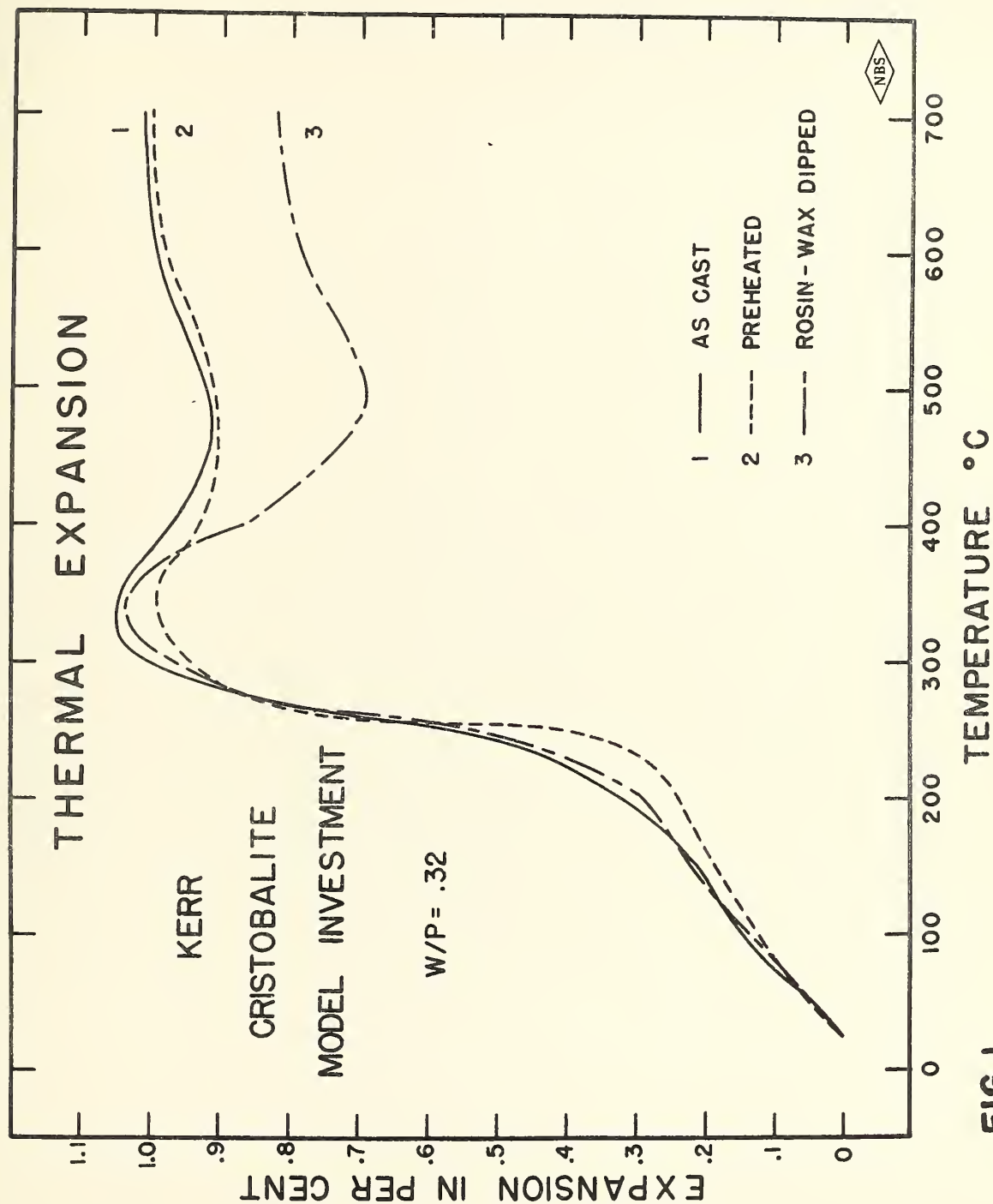


FIG.1

Figure 2

Thermal expansion of dental investment.

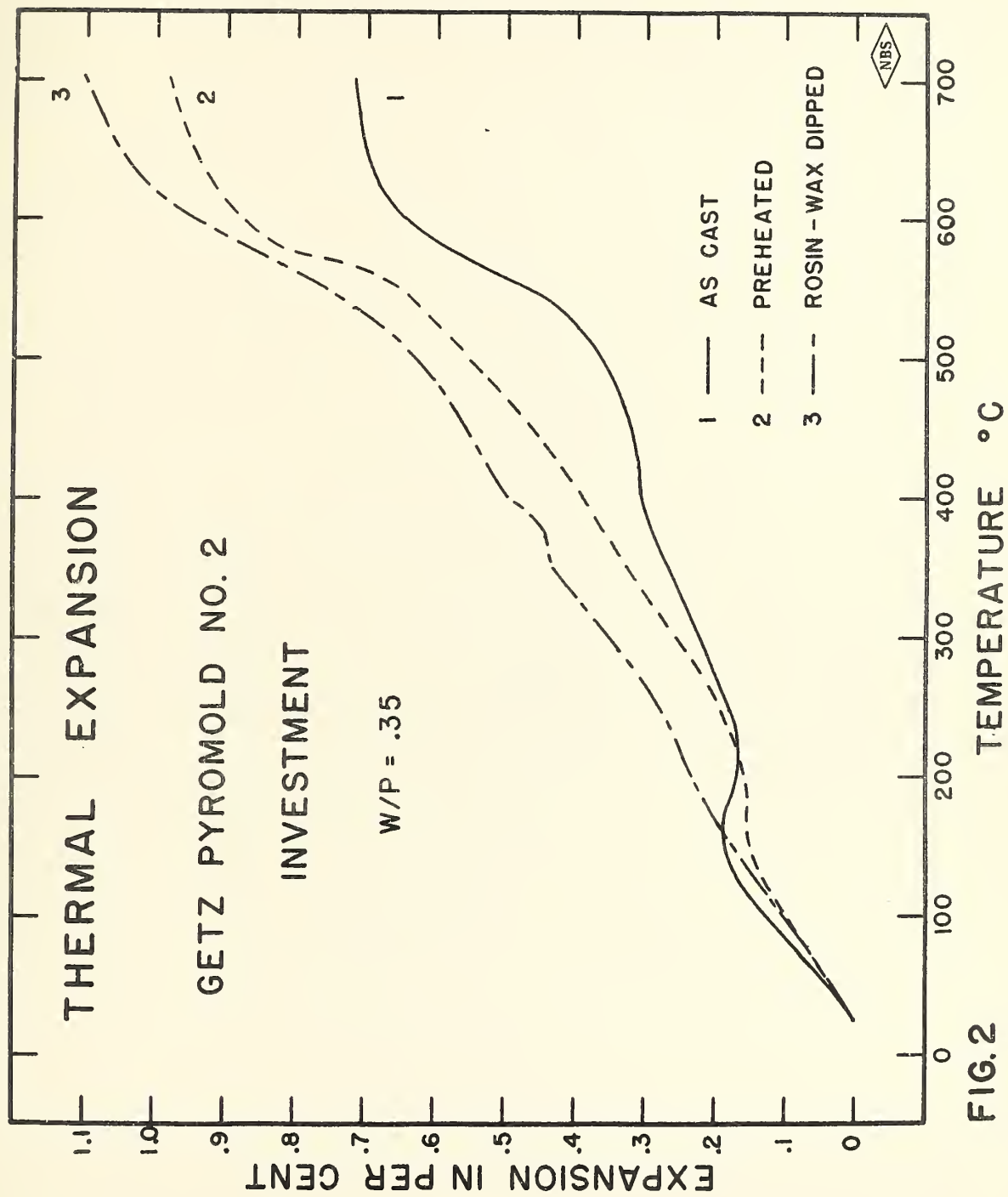


FIG.2

Figure 3

Thermal expansion of dental investment.

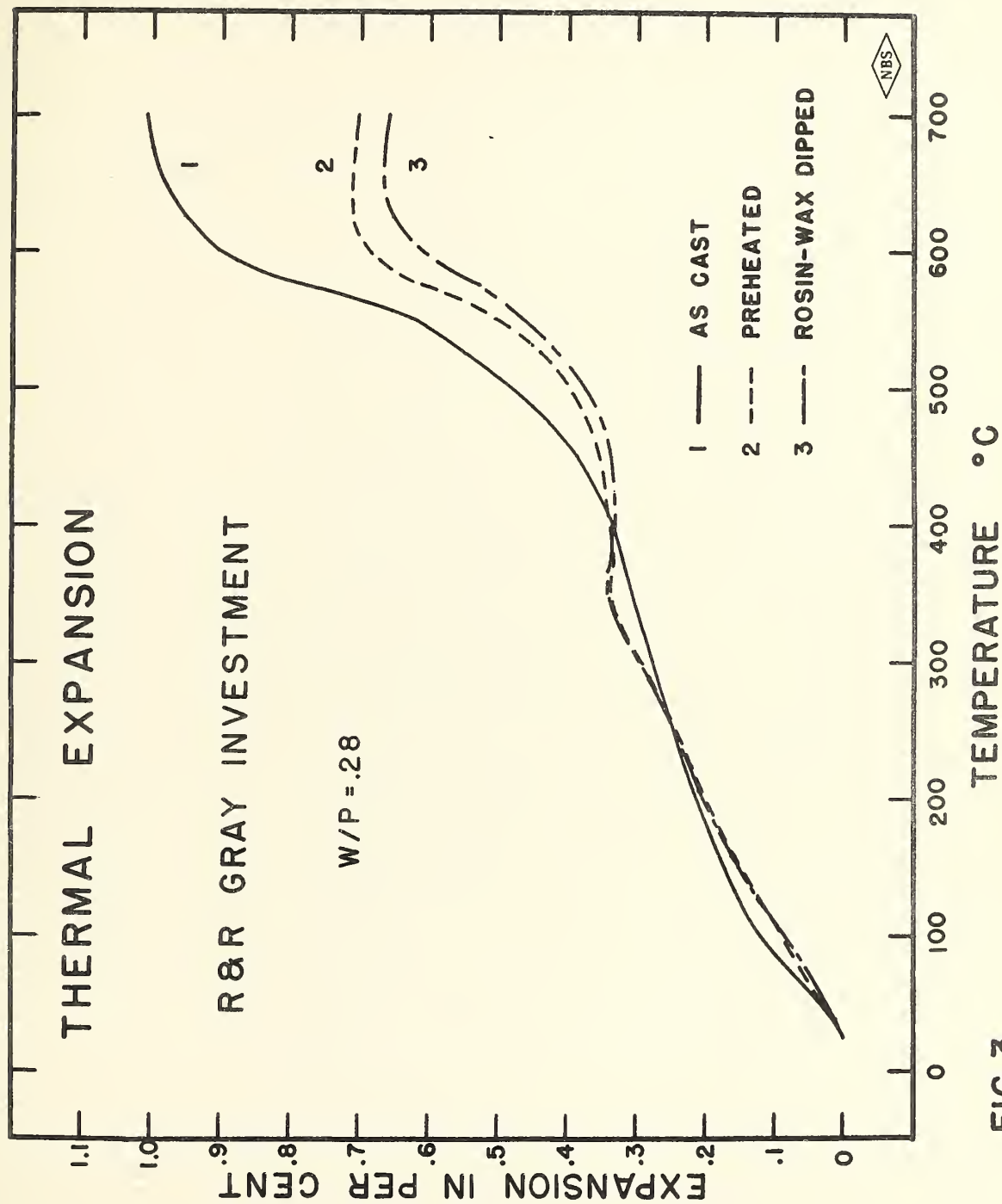


FIG. 3

Figure 4

Thermal expansion of gypsum.

THERMAL EXPANSION OF GYPSUM

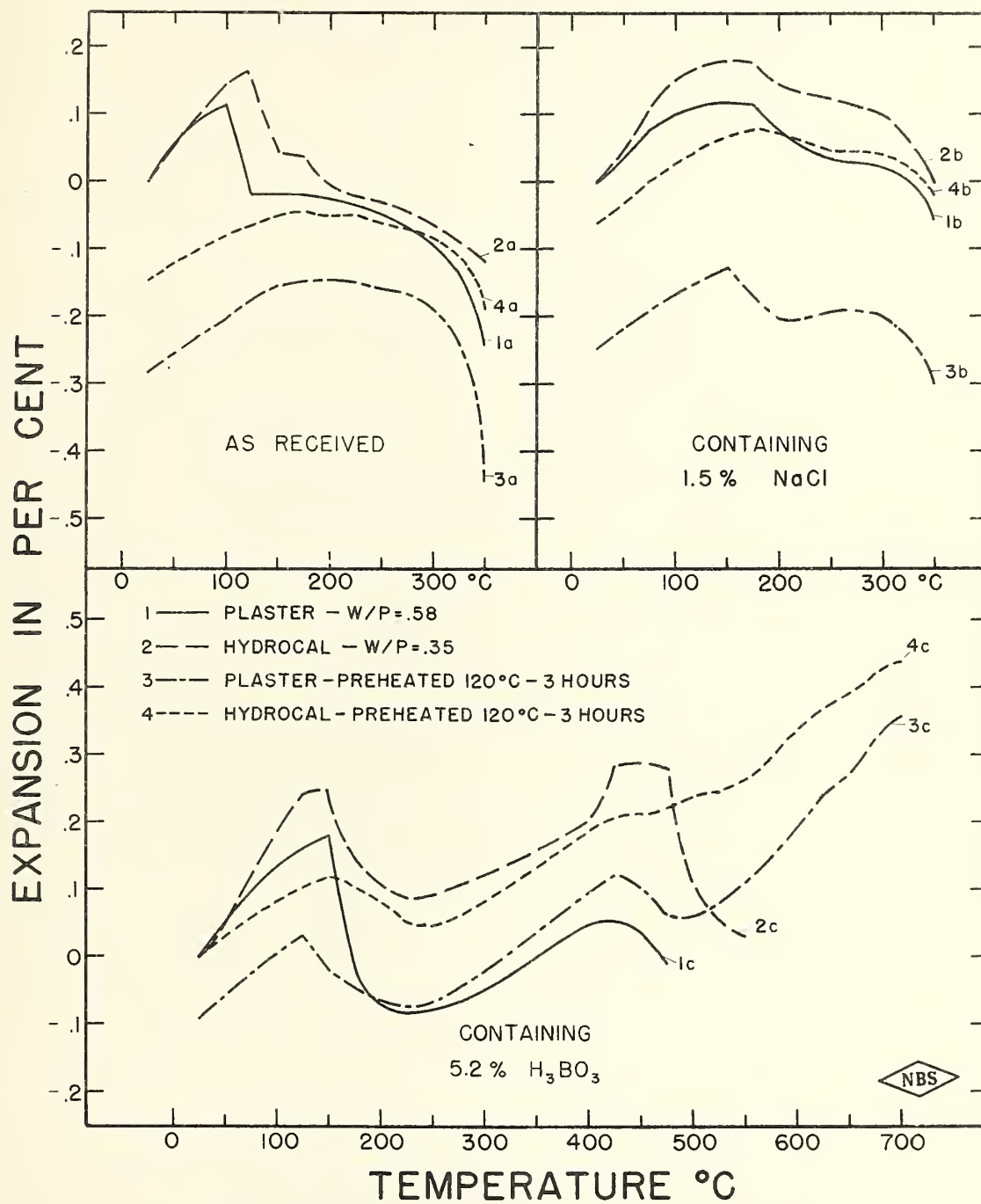


FIG. 4

Figure 5

Cross-section of partial denture mold which was heated to casting temperature. Note cracks at corners.

NBS

FIG. 5

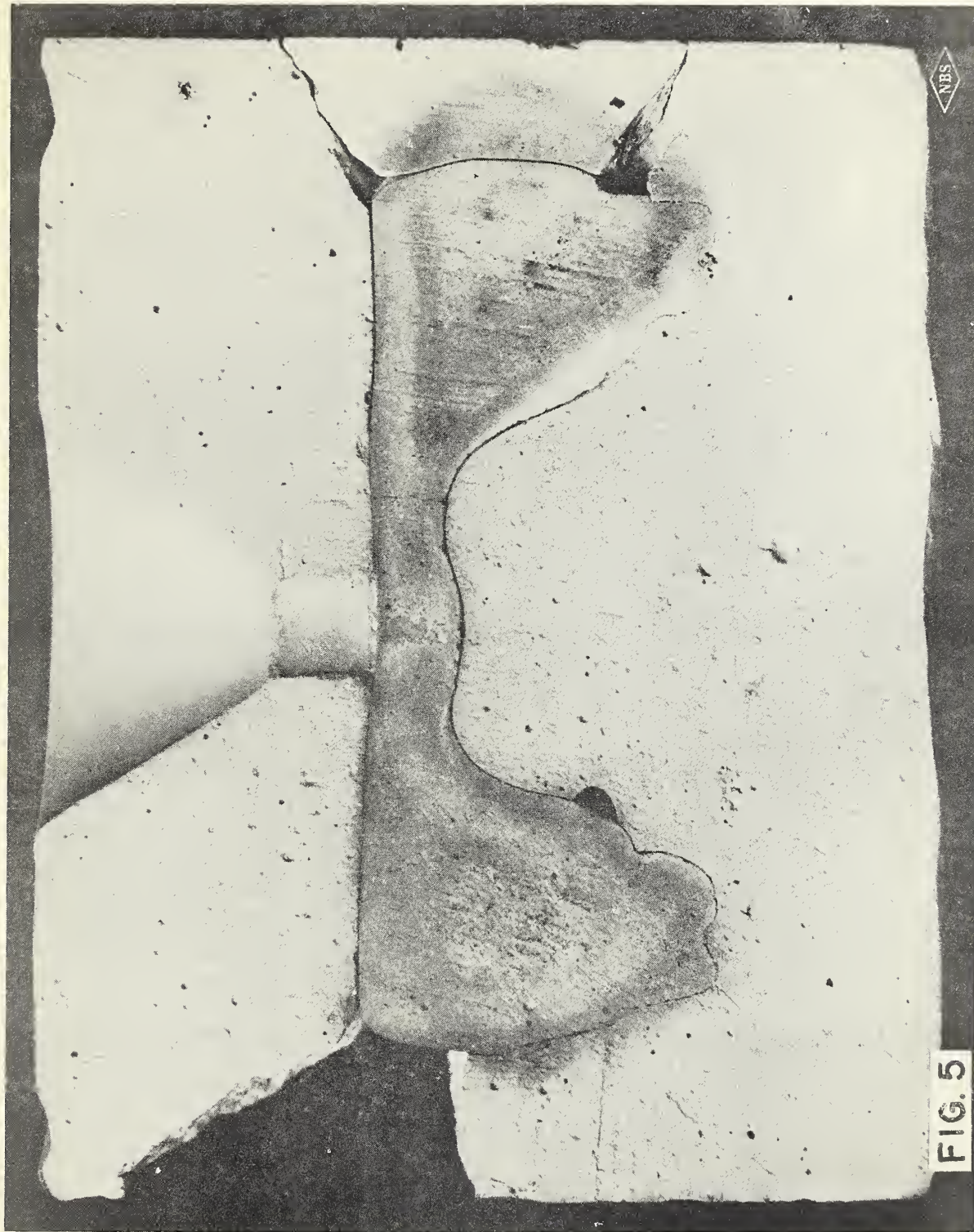


Figure 6

Technic castings made from identical wax patterns. Right shows satisfactory casting; left, typical fin formation resulting from use of high-expanding cast and low-expanding mold.



FIG. 6

Figure 7

Fit of M.O.D. inlays on steel dies; molds
given regular burnout. C, Cristobalite;
P, Pyromold; R, R & R Gray.

NBS

R

P

C

FIG. 7

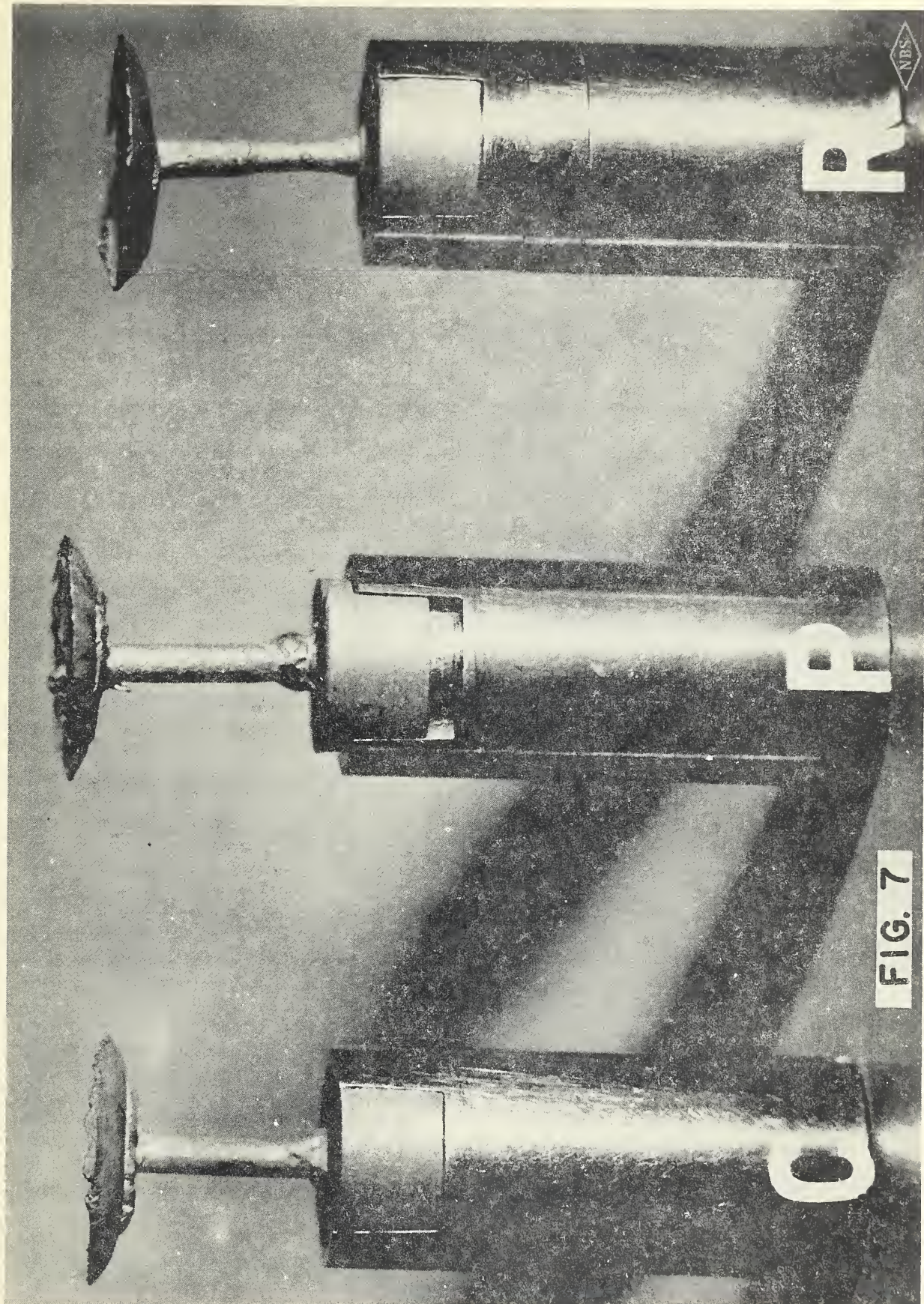


Figure 8

Fit of M.O.D. inlays on steel dies; molds given preheating treatment at 120° C and cooled to room temperature before regular burnout. C, Cristobalite; P, Pyromold; R, R & R Gray.

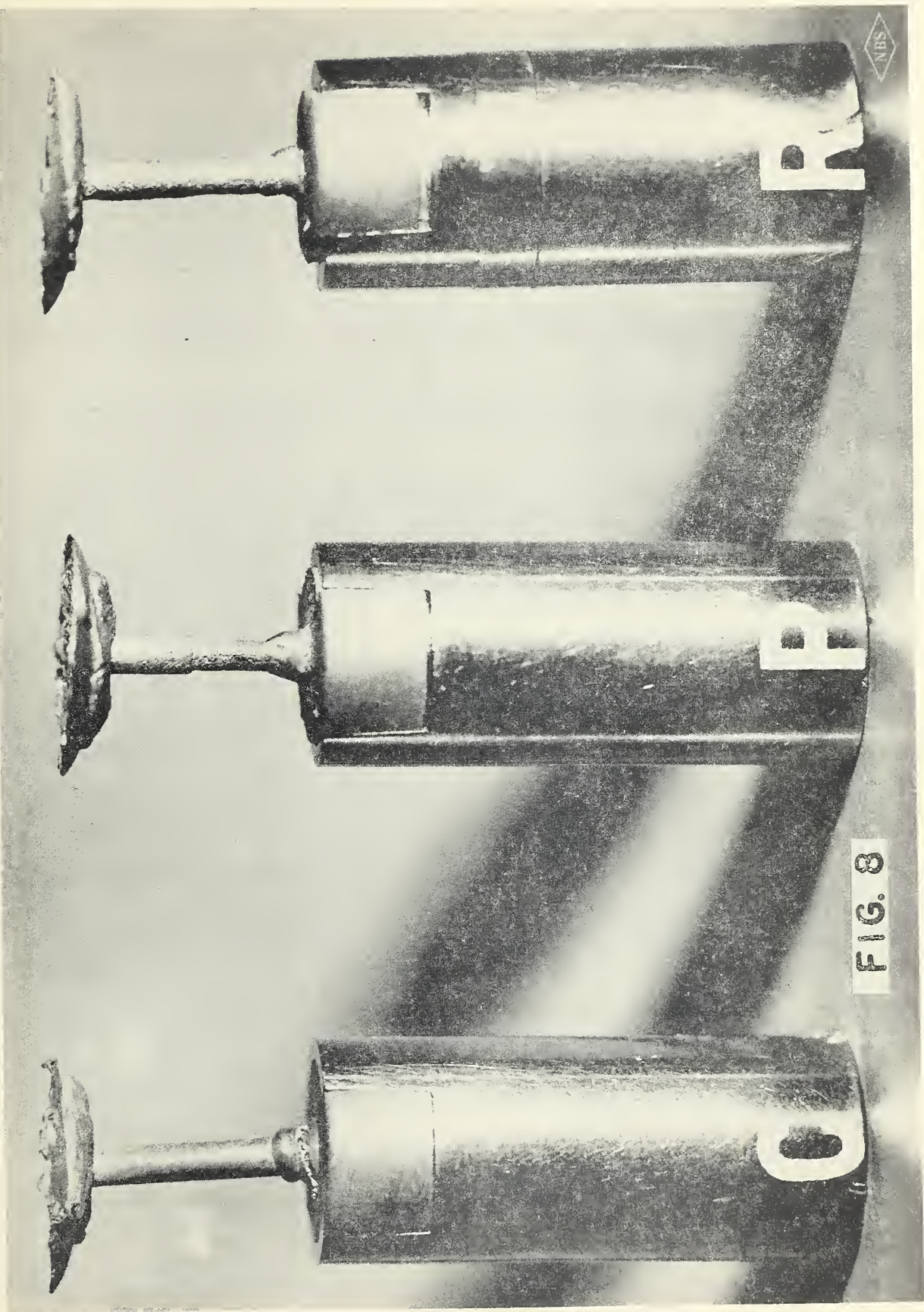


FIG. 8

Figure 9

Effect of preheating on thermal expansion
of three dental investments.

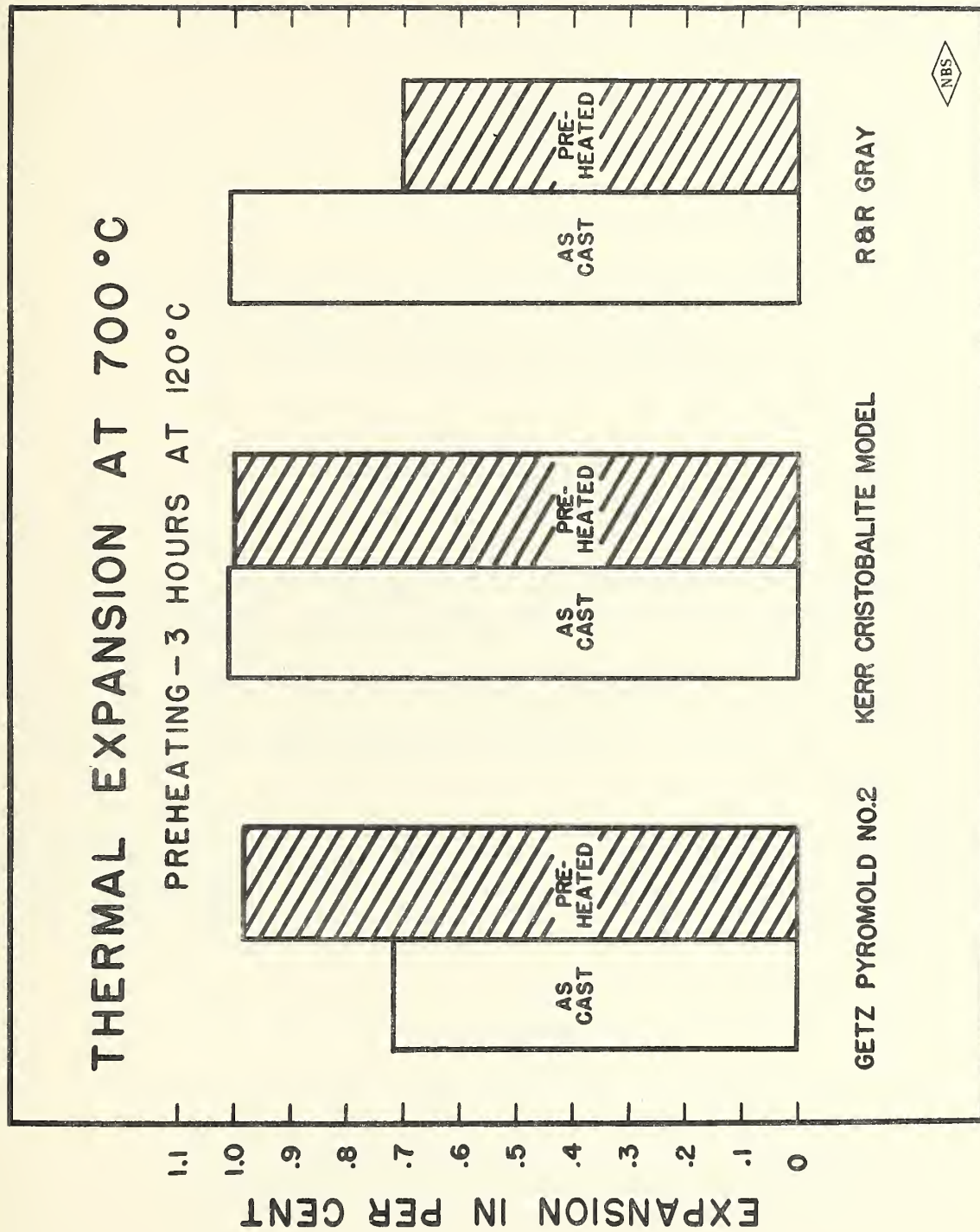


FIG. 9

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

NBS REPORT

0708-10-3824

February 3, 1953

2242

Progress Report

on

THE MECHANISM OF HYGROSCOPIC EXPANSION IN DENTAL CASTING INVESTMENTS

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THE MECHANISM OF HYGROSCOPIC EXPANSION IN DENTAL CASTING INVESTMENTS

Abstract

One of the methods used to produce the expansion of dental investments necessary to compensate for the casting shrinkage of gold alloys consists of placing the mold in water during the setting period. The mechanism of this hygroscopic expansion has not been definitely established. On the basis of the data presented it is believed that hygroscopic expansion of dental investments is a continuation of the normal setting expansion during the period when the investment is in a semisolid state. The introduction of additional water causes hygroscopic expansion by permitting further hydration of the calcium sulphate during this time. The inert filler present in the investment serves to weaken its structure and thus permit the precipitating hydrate crystals to expand the mass of material.

1. INTRODUCTION

One of the fundamental problems in restorative dentistry is the fabrication of cast gold appliances which accurately fit the cavity prepared in a tooth or the space between teeth located anywhere in the dental arches. Two important factors which must be considered in making such castings are the dimensional change of the wax pattern, and the contraction or casting shrinkage of gold on cooling from the liquid state to room temperature. At the present time there are two widely used technics that compensate for these shrinkages. Both depend upon expansion of the casting investment.

The older, more universal technic, often referred to as the "thermal expansion" method, depends upon the presence in the investment of a material or materials which, when heated, will expand in a known manner to enlarge the mold.

The more recently introduced method for increasing the mold size is based on the observation that some types of investment materials, when placed in water during the setting period, expand three to four times their normal setting expansion. The mechanism of this enlargement, commonly termed "hygroscopic expansion," will be discussed.

The phenomenon of hygroscopic expansion was reported by Wilson in 1920 [1] and was called to the attention of the dental profession by Scheu [2] in 1933. Scheu's original observations on the expansion of setting inlay investment upon the addition of water are the basis for the present hygroscopic technic. Scheu noted that all investments expanded hygroscopically and that the amount of expansion depended upon the water-powder ratio and time of immersion. Scheu defined hygroscopic expansion as that expansion occurring when water comes in contact with a mass of setting investment.

Numerous investigators [3,4,5,6] have published the results of their findings following Scheu's original observations. Degni [7] stated that the hygroscopic expansion was extremely sensitive to confinement of any kind. Degni also used the colloidal theory for the setting of plaster of Paris to explain the hygroscopic expansion process.

In a recent paper by Landgren [8] an excellent report on some of the factors affecting hygroscopic expansion was presented. Landgren noted that the water-powder ratio played an important role in determining the amount of expansion. He showed that the thicker the mix, the greater the amount of hygroscopic expansion. Landgren also showed that hygroscopic expansion decreases with an increase in time between spatulation and immersion when immersion is made after the initial set.

Generally speaking, the investigations mentioned above have been primarily concerned with measuring the amount of expansion which can be obtained hygroscopically and with determining the factors which affect such expansion. The mechanism of expansion has not been definitely established. It is the purpose of this paper to present a possible explanation of this phenomenon and to report some data supporting the explanation.

2. MECHANISM OF SETTING AND HYGROSCOPIC EXPANSION

2.1 Reactions Involved During the Normal Setting Expansion

Typical inlay investments contain approximately 30 percent gypsum (plaster of Paris or hydrocal) and 65 percent silica, the gypsum serving as a bonding medium for the silica. Plaster of Paris and the closely allied compound, dental stone or hydrocal, are essentially hemihydrates of calcium sulphate, $\text{CaSO}_4 \cdot \frac{1}{2} \text{H}_2\text{O}$. When the hemihydrate is mixed with water, the dihydrate $\text{CaSO}_4 \cdot 2 \text{H}_2\text{O}$, or gypsum is formed. The solubility of the hemihydrate is 0.65 grams per 100 milliliters of water and of the dihydrate 0.21 grams per 100 milliliters at 23.5°C [9]. Thus, when plaster is mixed with water, a nearly saturated solution of

the hemihydrate and a supersaturated solution of the dihydrate are quickly obtained. As the dihydrate is rapidly precipitated from solution, more hemihydrate is dissolved and crystallization continues. This process of solution and crystallization continues until the precipitated crystals have intermeshed so tightly that solidification occurs.

The hydrated gypsum formed in this process has a density of 2.32 [10] and a molecular weight of 172, giving a molar volume of 74.2 milliliters. Values varying from 2.55 to 2.75 have been reported for the density of the hemihydrate [9,10]. Since the hemihydrate has a molecular weight of 145, this gives a molar volume of approximately 55 milliliters. Thus, a volume increase of approximately 35 percent occurs on hydration. However, if the 27 milliliters of water required for this reaction are considered, a net shrinkage of 10 percent occurs. This shrinkage is not observed in a specimen of setting plaster. A slight expansion always occurs. The explanation of this apparent anomaly is based on the crystalline growth of the precipitated gypsum, which results in a porous structure with a greater apparent volume. Thus, the setting expansion observed in plasters results from crystal growth after and during the period when the mass becomes sufficiently solid to retain a definite form and just prior to the time at which the material attains sufficient strength to resist further deformation.

Apparently the hygroscopic expansion observed in investment materials results from an increased crystal growth over the period of time in which the mass is in a semisolid state and is a continuation of the normal setting expansion. The observations made during this investigation of the effect that inert substances and added water have on the amount of hygroscopic expansion appear to have a direct bearing on this proposed mechanism of continued expansion.

2.2 Effect of Inert Substances

The expansion observed when hydrocal is mixed with water is of the order of .04 to .06 percent. Hydrocal cannot be made to exhibit hygroscopic expansion. It was observed, however, that if an inert material was added to the hydrocal prior to mixing with water, the resulting mass invariably expanded hygroscopically. In these experiments the following inert materials were employed: silica, zinc dust, iron powder, acrylic resin powder, pumice, graphite, silica gel, sulfur, lamp black and talc. All the resulting mixes exhibited hygroscopic expansion. Some of the materials, particularly those difficult to wet, exhibited less expansion than others. Typical results obtained are shown in Table 1.

Table 1

Hygroscopic Expansion of Mixtures of
Hydrocal and Inert Substances

Substance Added	Setting Expansion	
	Inert substance 20 percent by weight	Inert substance 60 percent by weight
	percent	percent
Pumice	.4	.75
Graphite	.7	1.6
Talc	.8	2.8
Silica Gel	3.1	8.1

It appears that the effect of the inert material was to weaken the structure of the mass to such an extent that expansion resulting from crystalline growth could continue for a longer period of time before the mass gained sufficient strength to resist further deformation. Such crystalline thrust or growth is not uncommon in the solidification of metals from the liquid state [11] .

In addition to loosening the structure of the setting investment, the presence of inert materials may allow more efficient penetration of water into the structure of the investment mass.

2.3 Effect of Water

To make an investment expand hygroscopically, additional water must be made available to the investment during a critical period in the hardening of the mass. This need for additional water is difficult to explain, particularly because of the fact that water in excess of that required for complete hydration is always added in making the initial mix. The following explanation for this need of additional water is proposed.

In the setting reaction of investments crystallization does not occur uniformly throughout the mass. During this nonuniform crystallization, powder particles begin to dissolve and crystals of the hydrate grow on the surface of the undissolved hemihydrate. Further crystalline growth causes water to be retained in the voids in and around the crystallites. Thus, although sufficient water is present initially to hydrate all the hemihydrate, it is temporarily prevented from doing so by this entrapment. Eventually, by a process of

diffusion of the water or water vapor, complete hydration will occur. However, the process is slow and the mass will have gained sufficient strength to resist deformation before hydration is completed.

If the investment is placed in water prior to such hardening, the water rapidly penetrates the mass as a result of capillary action. Additional hemihydrate is dissolved, dihydrate is precipitated, and further crystallization continues with sufficient rapidity to expand the semisolid mass. That some such mechanism is in operation is made evident by the rapidity with which water is imbibed by the mass during this critical period and its immediate effect in expanding the mass. This was demonstrated by placing a cylindrical specimen of investment 1.25 inches in diameter and 1.5 inches high under a dial gage. At the critical period during which hygroscopic expansion occurs most readily, drops of water were applied to various areas over the surface of the specimen by means of a medicine dropper. An immediate increase in length was observed as each drop of water was added.

In other experiments a small amount of water was placed around the base of a specimen. When the setting reaction reached the critical period for hygroscopic expansion to occur, the water was rapidly pulled into the specimen and expansion followed immediately.

2.4 Volume Changes During Hydration

If the proposed mechanism of further crystallization is valid, some of the hemihydrate must remain unhydrated during the period in which the setting mass is most susceptible to hygroscopic expansion. That hemihydrate is present in the mix during this period was demonstrated by an experiment in which the rate of hydration was observed by measuring the decrease in volume of a mixture of investment and water containing a large excess of water. The investment powder was inserted into an apparatus so designed that all air could be pumped out of the powder mass and water added as shown in Figure 1.

Sufficient water was added to cover the specimen and rise in a graduated capillary tube joined to the specimen chamber. The apparatus, sample, and water were in an air bath maintained at a constant temperature to within $\pm 0.1^\circ\text{C}$. The height of the water in the capillary tube was recorded at intervals from eight minutes to three hours after the water was admitted to the specimen chamber.

The results obtained on several brands of investment and one dental artificial stone are shown in Figure 2. The data show that shrinkage of the investment-water mixtures continued at a rapid rate for approximately one hour after the investment and water were mixed. This indicates that hemihydrate is present in the mix and that hydration continues during the time that hygroscopic expansion occurs. It is reasonable to believe, therefore, that water added to the investment causes hygroscopic expansion to occur by inciting a more complete hydration while the material is in a semisolid state. It is possible that the water added weakens the structure of the setting investment and thus permits a greater expansion of the investment by the growth or thrust of the precipitating hydrate crystals.

2.5 Effect of Retarders and Accelerators

If hygroscopic expansion is only an extension of setting expansion as a result of sorption of water during a critical period in the setting of gypsum, as proposed, materials which retard the hydration and hardening of gypsum would be expected to lengthen this critical period, thus permitting more sorption of water and causing an increase in the hygroscopic expansion of investments. To test this, cylindrical specimens of an investment to which a retarder had been added were prepared. These cylinders were placed under a dial gage as soon as they possessed sufficient strength to support themselves. Water was then poured into the container surrounding the investment and the amount of expansion was measured on the dial gage. Similar experiments were made on specimens containing no retarder. The increased amount of expansion in specimens containing retarders is shown in Table 2. The retarders apparently prevented rapid hydration and allowed greater crystalline growth in a weakened structure.

The experiment was repeated substituting accelerators for retarders. The extreme rapidity of set resulted in a much lower expansion. Probably because of the increased speed of hydration and the rapid interlocking of the crystals into an extremely hard mass in a short period of time, the amount of expansion was not affected by immersion in water.

2.6 Production of Hygroscopic Expansion by Silica Gel

Based on the principles demonstrated in the experiments on the effect of inert substances and water on hygroscopic expansion, an experimental investment was prepared using silica gel previously saturated with water and air dried, mixed with hydrocal. Specimens of this investment containing 50 percent hydrocal and 50 percent silica gel expanded

approximately 3 percent without the addition of water. The amount of expansion of other specimens containing different ratios of hydrocal to silica gel was roughly proportional to the amount of silica gel added. It is postulated that the silica gel, capable of holding up to 90 times its own weight of water, acted both as an inert material and as a source of water for the continued hydration of the hemihydrate.

Although these experimental investments behaved in accordance with the proposed mechanism for hygroscopic expansion, they were not satisfactory for practical use in an inlay technic.* However, this preparation of an investment which will expand hygroscopically without the addition of water suggests the possibility of the development of a satisfactory dental investment based on this principle.

Table 2

Effect of Accelerators and Retarders on Hygroscopic Setting Expansion of Cristobalite Investment

Material Used	Action	Setting Time	Expansion
		min	percent
Crystobalite investment + ferrous sulphate, 2 percent; wet asbestos liner; not immersed	Accelerator	10	.6
Crystobalite investment + ferrous sulphate, 2 percent; wet asbestos liner; immersed	Accelerator	10	.6
Cristobalite investment, wet asbestos liner; not immersed	None	25	1.0
Cristobalite investment; freely expanding; immersed	None	25	1.5
Sodium acetate, 4 percent; wet asbestos liner; not immersed	Retarder	90	1.2
Sodium acetate; freely expanding; immersed	Retarder	120	2.2

*Heating these investments caused shrinkage and reduced strength. Castings made in unheated molds were unsatisfactory, apparently because of the vaporization of water in the investment by the molten gold.

3. CONCLUSIONS

On the basis of the data presented it is believed that hygroscopic expansion of dental investment is a continuation of the normal setting expansion during the period when the investment is in a semisolid state. This continued expansion is produced by the introduction of additional water which permits further hydration of calcium sulphate at this time. The inert filler present in the investment weakens its structure and thus permits the precipitating hydrate crystals to expand the mass of material.

Bibliography

1. Wilson, G. H. Manual of Prosthetic Dentistry, p. 78 (Lee and Febiger, Philadelphia, 1920).
2. Scheu, C. H. "A New Precision Casting Technic." J. Am. Dent. Assn. 19: 630 (1933).
3. Smyd, E. S. "Factors Which Influence Casting Accuracy: A Universal Casting Technic." J. Am. Dent. Assn. 36: 160-172 (1948).
4. Docking, H. R. "The Hygroscopic Setting Expansion of Dental Casting Investments, Parts I, II and III." Austral. J. Dent. 52: 6-11; 160-166; 320-329 (1948).
5. Hollenback, G. M. "Precision Gold Inlays Made by a Simple Technic." J. Am. Dent. Assn. 30: 99-109 (1943).
6. Coy, H. D. "Important Factors in Making Satisfactory Small Castings." N.Y. J. Dent. Vol. XX, No. 3, p. 105-109.
7. Degni, F. "Hygroscopic Expansion of Dental Investments." Thesis, Northwestern University Dental School.
8. Landgren, N. "Hygroscopic Expansion of Some Casting Investments." Thesis, University of Michigan School of Dentistry.
9. Kelly, K. K., Southard, J. C., and Anderson, C. T. "Thermodynamic Properties of Gypsum and Its Dehydration Products." Bureau of Mines Technical Paper 655 (1941).
10. Winchell, A. N. Microscopic Character of Artificial Minerals p. 226-229 (John Wiley & Sons, New York, 1931).
11. "Method for Obtaining High Pressures at Low Temperatures." J. Physics (USSR) 8: 193-200; J. Exper. & Theor. Physics (USSR) 14: 439-47 (1944).

Figure 1

Apparatus used to investigate rate of hydration of gypsum in water by measurement of decrease in volume resulting from hydration. The gypsum is placed in the specimen chamber at the bottom of the apparatus. After this chamber has been evacuated, water from the spherical reservoir is allowed to flow around the specimen. The reservoir is then sealed off, the appropriate capillary tube is opened, and the change in volume is observed during the period of hydration of the specimen.

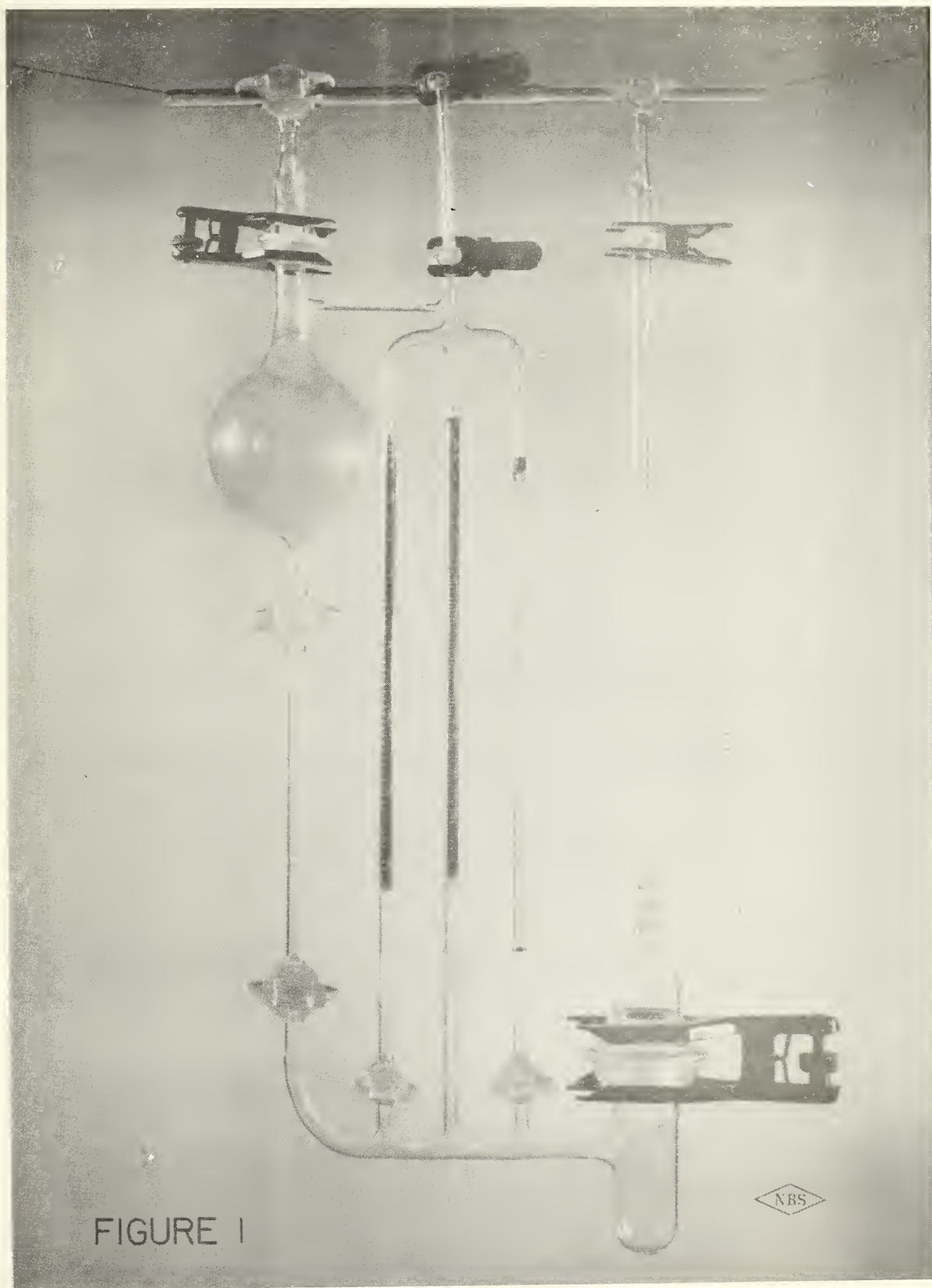


Figure 2

Rate of hydration of dental investments and stone as indicated by change in volume of water and investment or stone mixture. Change in volume of the mixture is indicated as milliliters of water.

RATE OF HYDRATION OF DENTAL INVESTMENTS

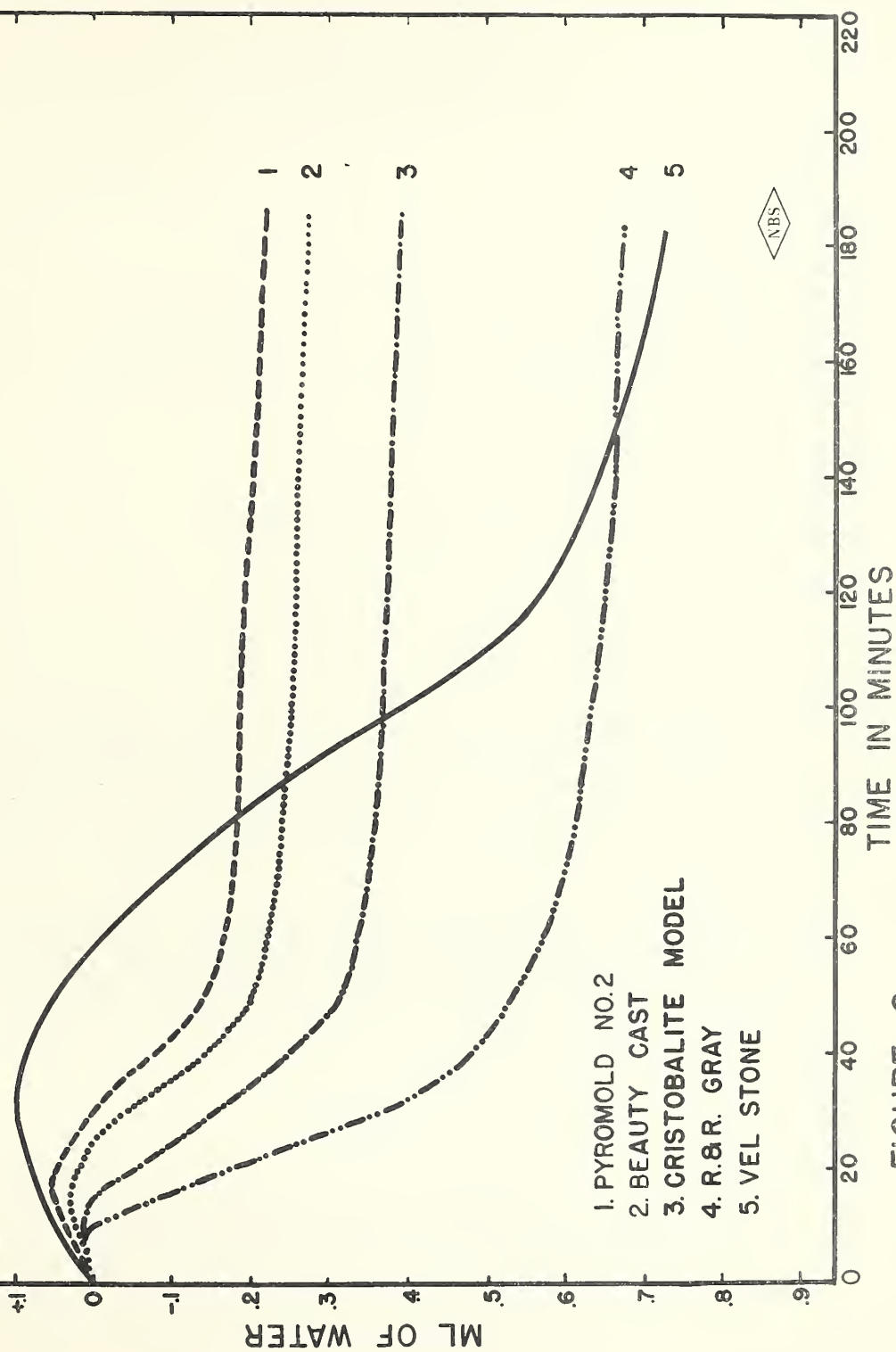


FIGURE 2

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NBS REPORT

0708-10-3824

February 15, 1953

2283

Progress Report
on
TEMPERATURES DEVELOPED IN
ROTATING DENTAL CUTTING INSTRUMENTS

by

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This work is a part of the dental research program conducted at the National Bureau of Standards in cooperation with the Council on Dental Research of the American Dental Association, the Army Dental Corps, the Air Force Dental Service, the Navy Dental Corps and the Veterans Administration.



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TEMPERATURES DEVELOPED IN ROTATING DENTAL CUTTING INSTRUMENTS

Abstract

The Air Force has been interested in the problem of heat trauma in dental operating as a possible cause of pain occurring in teeth at high altitudes. A direct approach to the problem of bur temperature measurements has been undertaken, placing a thermocouple in the head of the bur. Behavior of dental burs of steel and carbide has been studied at speeds up to 24,000 r.p.m. Temperatures ranging from 149 to 275°F were recorded when cutting natural dentin and a synthetic substitute. Cutting rates were also determined over the same speed range, and were found to be proportional to the speed of rotation of the bur. Results of the work indicate that temperatures well above the limit of tolerance of tooth structures to heat are attained by the cutting bur. A coolant would appear to be necessary if unfavorable reactions are to be prevented when operating dental burs at high speeds.

An Air Force survey conducted by some 200 dental officers in 1946 revealed that large numbers of flying personnel were afflicted with dental pain when subjected to low atmospheric pressures [1]. This pain was of such severity as to interfere with the performance of duty as members of an air crew. Most of these painful conditions were found to occur in recently filled teeth, and it was suspected that the trauma of cutting, and the heating of tooth structure which occurs during cavity preparation, were responsible for the ill effects.

A project was started at the School of Aviation Medicine at Randolph Field, Texas, at that time, to determine some of the factors causing trauma to dental pulps when preparing cavities by means of rotating cutting instruments. This project was resumed at the National Bureau of Standards in 1950 under a grant from the School of Aviation Medicine.

A review of the dental literature bearing on the subject of heat production and cutting efficiency of burs, and the traumatic effects of cavity preparation and pulp reactions to heat was conducted. Work by Jeserich [2], Bodecker [3], Walsh and Simmons [4], Henschel [5,6,7], Anderson and Van Praagh [8], Lisanti and Zander [9], and Peyton and Vaughn [10], led to several conclusions which formed the basis for the study carried out at the National Bureau of Standards.

These conclusions were: (a) The upper physiological tolerance of normal dentin to heat lies in the range 85° to 130°F [5]. (b) Application of heated instruments in the range 125° to 600°F to the floor of cavity preparations in dogs' teeth for varying lengths of time caused definite alterations in the odontoblastic layer, blister formation and inflammatory changes [9]. (c) Heat production by revolving burs increases with increasing speed of rotation [10]. (d) Cutting rate increases with an increase in speed and pressure [10].

Previous workers used indirect methods, e.g. calorimetric determinations of heat production during burring, to determine bur temperature [7]. Others measured temperatures by means of thermocouples inserted in teeth at points adjacent to that being cut by the revolving bur [10]. Estimates of bur temperature were also made, based upon such things as observed changes in color of the steel, and the melting of tin-lead alloys when cut by dental burs [7].

A more direct approach to the problem of determining bur temperature was attempted in the study reported here. Efforts were directed toward the measurement of the temperature of the head of the bur itself during cutting operations.

APPARATUS AND METHODS

A machine designed to rotate dental burs at speeds up to 25,000 r.p.m., with accessory timing and controlling features, constructed at the School of Aviation Medicine, was shipped to the National Bureau of Standards at the time of the project transfer. It was modified so that the following conditions could be accurately controlled: (a) speed of rotation up to 25,000 r.p.m. to ± 200 r.p.m.; (b) depth of cut to $.15 \pm 0.001$ inch; (c) cutting load to 600 ± 1 gram. Provision for accurate recording of time to 1/1000 minute was included in the apparatus, as was control of the starting and stopping of the cutting cycle by means of electromagnetic devices. All burring was done with 2.35 mm, #8 eight-bladed round burs cutting axially, mounted in a vertical precision chuck. Specimens to be cut were lowered from above by gravity and weights were placed on the spindle to exert the desired pressure on the bur (Fig. 1).

A constantan wire, #36, 0.005 inch in diameter, was soldered in the groove between the blades of each test bur (Fig. 2). With the aid of a binocular microscope, the excess solder was cut away with a fine instrument to prevent its interfering with the operation of the bur. The wire was secured to the bur shank with an insulating adhesive until it reached the slip ring commutator to which it was connected (Fig. 3). The circuit was completed through the bur itself and another commutator ring at the base of the chuck.

The e.m.f. generated in the bur-constantan combination was picked up by brushes connected to a Brown Instrument recording potentiometer calibrated in degrees centigrade. The bur-constantan combination was calibrated and a curve obtained which was used to correct the readings of the recording thermometer.

RESULTS

Human tooth enamel and dentin were first cut holding the tooth by hand and exerting sufficient force to achieve normal cutting as in operating. The temperatures recorded varied from 150°F at 2,000 r.p.m. to 275°F at 7,500 r.p.m. when cutting dentin alone. When the bur was moved laterally to cut both enamel and dentin, as in the extension of a cavity preparation, temperatures as high as 350°F were observed.

An effort was made to penetrate the occlusal enamel of a molar tooth at 7,500 r.p.m. The bur was engaged in a non-carious occlusal pit, and force such as would be exerted in normal operating was applied. Cutting was very slight and the temperature of the bur mounted rapidly, reaching an indication of over 600°F. Examination of the bur revealed dulling of the blade edges (Fig. 4-A), and marked color changes were noted. The blue color developed corresponds to that indicated for steel temper colors at temperatures near 600°F [11].

Reproducibility of results was unsatisfactory due to the wide variations in properties of natural tooth substance from tooth to tooth. Therefore, it was decided that a synthetic substance that was cut by the bur at approximately the same rate as was dentin, would be used for all temperature and cutting rate measurements. The material chosen was a cast, filled Bakelite resin (P-183) from which conveniently sized specimens were prepared.

The curve shown in Fig. 5 is plotted from data obtained in a series of ten test cuts in this material at a load of 200 grams and at speeds from 2,000 to 24,000 r.p.m. (Table 1). All cuts in each run were made to a depth of 0.15 inch, and the temperatures attained by the bur are shown to approach a maximum at a speed of 6,000 r.p.m. As the speed increased beyond this point, there was little change in temperature. A series of five cuts was made at a load of 200 grams, with the constantan wire soldered on the slope of the bur blade rather than in the groove between blades. It will be noted from Table 1 and Fig. 6 that the temperatures recorded at points on the blade were higher than those taken at points farther from the edge (in the groove).

The time required to complete a fixed depth of cut, plotted against rotational speed is shown in Fig. 7. The data are shown in Table 1. It is interesting to note that the time necessary to complete the fixed depth cut falls rapidly as the speed increases, until the region 8,000 to 12,000 r.p.m. is reached. Beyond this point the curve flattens and the time required to complete the cut at 24,000 r.p.m. is only about 3 seconds less than that required at 10,000 r.p.m., whereas the difference in time required to make the cut between 2,000 and 12,000 r.p.m. is as much as 24 seconds. These data indicate within the limits of experimental error that steel and carbide burs cut faster as speed of rotation increases up to about 20,000 r.p.m. Practically, there would not appear to be any great advantage as far as rate of cutting is concerned, in increasing speeds of rotation above approximately 12,000 r.p.m. The time required for cutting at this speed is so low that further reduction would probably not decrease significantly the time required for the preparation of a cavity under actual operating conditions.

The above experiments were repeated, using steel-shanked, carbide-tipped burs of the same size and blade arrangement as those of the all-steel burs. A curve of temperature versus revolutions per minute, plotted from data given in Table 1, is shown in Fig. 8. The time required to reach a fixed depth in cutting is given in Table 1, and the curve is shown in Fig. 9. It will be noted that the rate of cut compares closely with that of the all-steel bur.

Carbide-tipped burs penetrated tooth enamel easily without damage to the bur blades (Fig. 4-B).

Lisanti and Zander have shown that instruments heated to temperatures in the range 125-600°F and applied to the pulpal floors of cavities in normal dogs' teeth produced definite pulpal reactions [9]. It would be reasonable to assume, therefore, that dental operative procedures performed with burs that reach temperatures of from 140° to 250°F may be responsible for unfavorable reactions in the human tooth. Postoperative pain occurring at high altitudes in recently restored teeth of flying personnel may be due to volume changes in blisters or areas of separation of the odontoblastic layer from the pulpal wall with variations in barometric pressure. The use of a coolant would appear to be indicated, especially when operating at high speeds.

CONCLUSIONS

1. The temperatures reached by the dental bur in cutting natural dentin ranged from 150° to 275°F. Temperatures attained when using a synthetic substitute ranged from 140° to 250°F measured on the slope of the bur blade. Since these values are

above those said to be tolerated by normal human dentin [5], it would seem advisable to use some form of coolant when burring, especially at high speeds.

2. Steel and carbide burs cut more rapidly as their speed of rotation increases up to about 20,000 r.p.m. under the conditions used in this investigation.

3. Steel burs are not suited to the cutting of tooth enamel because of their relative softness, whereas carbide burs will cut readily.

REFERENCES

1. Preliminary Report, School of Aviation Medicine, 31 December 1949.

2. Jeserich, P. H. Factors Necessary to Minimize Thermal Changes in Tooth Structure from Operative Procedures, N. Y. J. Dentistry V, 275-278 (1935).

3. Bodecker, C. F. Demonstration of Possible Ill Effects of Heat on the Pulp, Caused by Rapid Operating Technic, J. Am. Dent. Assn. 26: 527-532 (1939).

4. Walsh, J. P., and Simmons, H. F. Comparison of the Heat Production and Mechanical Efficiency of Diamond Instruments, Stones and Burs at 3,000 and 60,000 R.P.M., New Zealand Dent. J. 45: 28-32 (1949).

5. Henschel, C. J. Development of Thermal Control, J. Am. Dent. Assn. 33:194-200 (1946).

6. Henschel, C. J. Heat Impact of Revolving Instruments on Vital Dentin Tubules, J. Dent. Res. 22:323 (1943).

7. Henschel, C. J. The Friction of Revolving Steel Burs, J. Am. Dent. Assn. 31:895 (1944).

8. Anderson, D. J., and Van Praagh, G. Preliminary Investigation of the Temperatures Produced in Burring, Brit. Dent. J. 73:62-64 (1942).

9. Lisanti, V. F., and Zander, H. A. Thermal Injury to Normal Dog Teeth: In Vivo Measurements of Pulp Temperature Increases and Their Effect on the Pulp Tissues, J. Dent. Res. 31: 548-558 (1952).

10. Peyton, F. A., and Vaughn, A. B. Thermal Changes Developed During the Cutting of Tooth Tissue. Paper presented at the Mid-Winter Meeting of the Chicago Dental Society, February 1950.

11. Young, C. B. F. Coloring Steel, Metals Handbook, p. 730 (1948 Edition).

Table 1

Effect of Rate of Rotation on Temperature Rise
and Cutting Rate of Dental Burs*

Rotation	STEEL BURS			CARBIDE BURS	
	Temperature		Time sec	Temperature	Time sec
	In Groove °F	On Blade °F		In Groove °F	
r.p.m.					
2,000	145 ± 3	142 ± 3	28.7 ± 7.1	162 ± 4	28.6 ± 8.0
4,000	160 ± 8	212 ± 8	14.4 ± 4.0	175 ± 4	8.3 ± 1.0
6,000	178 ± 4	232 ± 11	8.1 ± 2.4	184 ± 2	6.8 ± 1.0
8,000	181 ± 4	237 ± 13	5.7 ± 1.4	192 ± 2	5.5 ± 1.0
10,000	179 ± 8	239 ± 18	4.3 ± 0.7	196 ± 4	4.6 ± 1.0
12,000	185 ± 4	243 ± 12	3.6 ± 0.2	198 ± 2	4.0 ± .0
14,000	179 ± 6	244 ± 16	2.9 ± 0.4	198 ± 3	3.0 ± .0
16,000	186 ± 5	248 ± 11	2.3 ± 0.2	198 ± 2	2.5 ± .2
18,000	185 ± 5	246 ± 12	2.1 ± 0.2	196 ± 2	2.3 ± .1
20,000	181 ± 6	242 ± 6	1.8 ± 0.2	194 ± 2	2.0 ± .1
22,000	182 ± 6	243 ± 8	1.7 ± 0.1		
24,000	182 ± 6	245 ± 9	1.7 ± 0.1		

*Data obtained while cutting synthetic dentin substitute (Bakelite Cast Resin #P-183), with #8 round burs. Force of 200 grams applied, burs cutting axially to depth of $0.15 \pm .001$ inch. Thermocouple wire applied to groove between blades and on slope of blade in the case of steel burs. Thermocouple wire applied in groove between blades in the case of carbide burs.

Figure 1

Bur evaluating machine. Vertical spindle with chuck, driven by variable speed motor, along with accessory timing and limiting controls.

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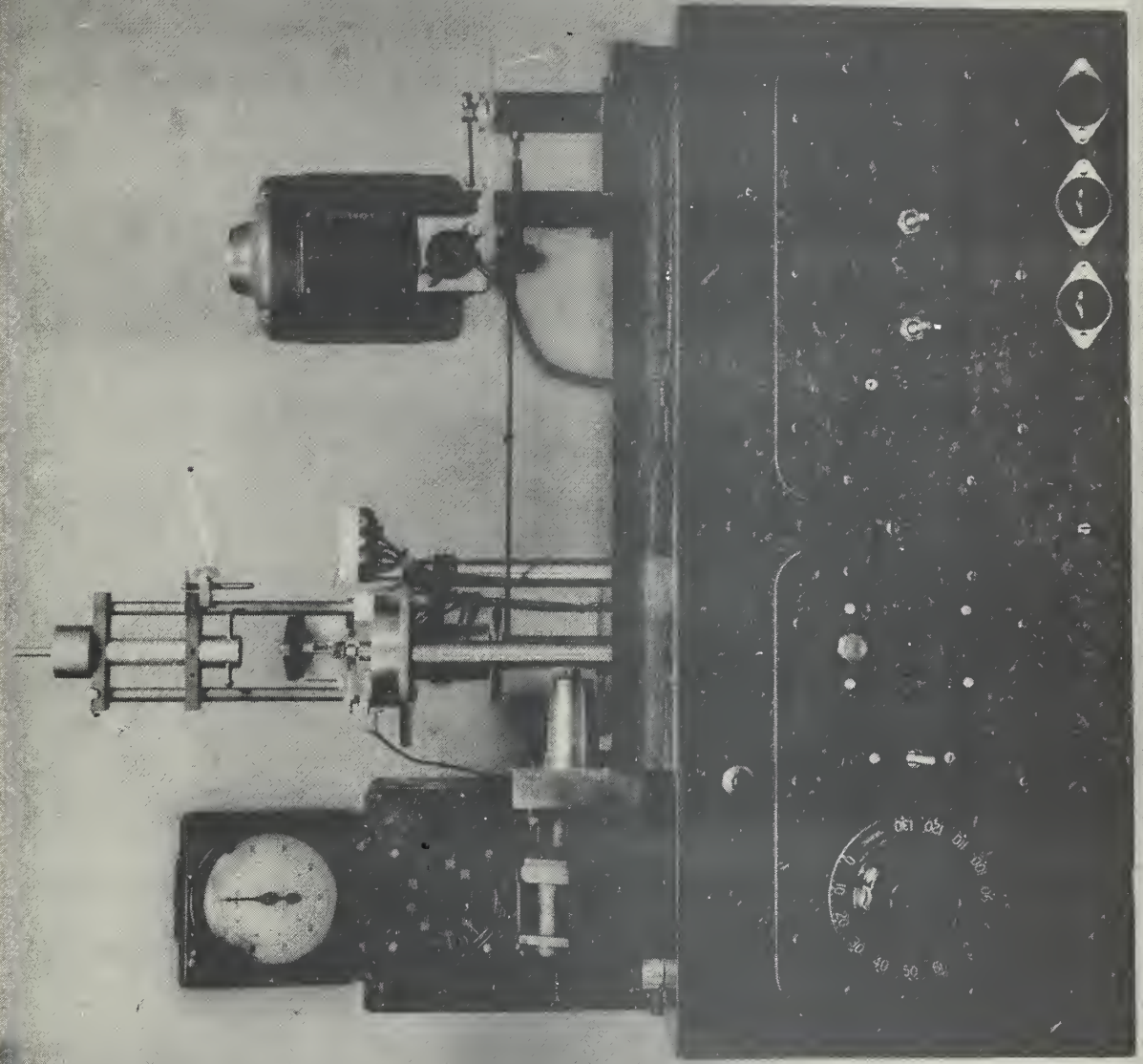


Figure 2

Thermocouple wire soldered in groove between
bur blades.



Figure 3

Bur-holding chuck, showing commutator and brush assembly.

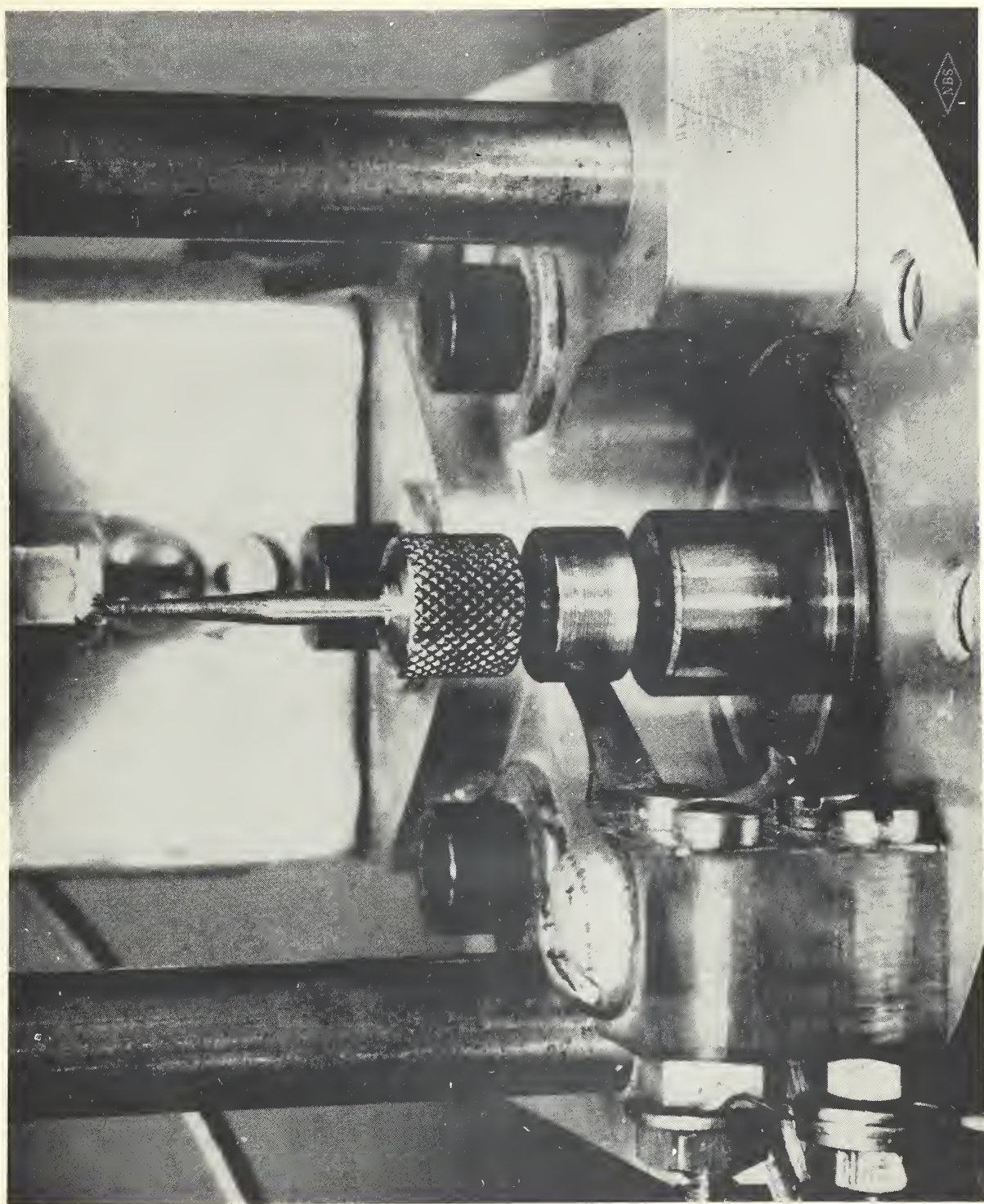
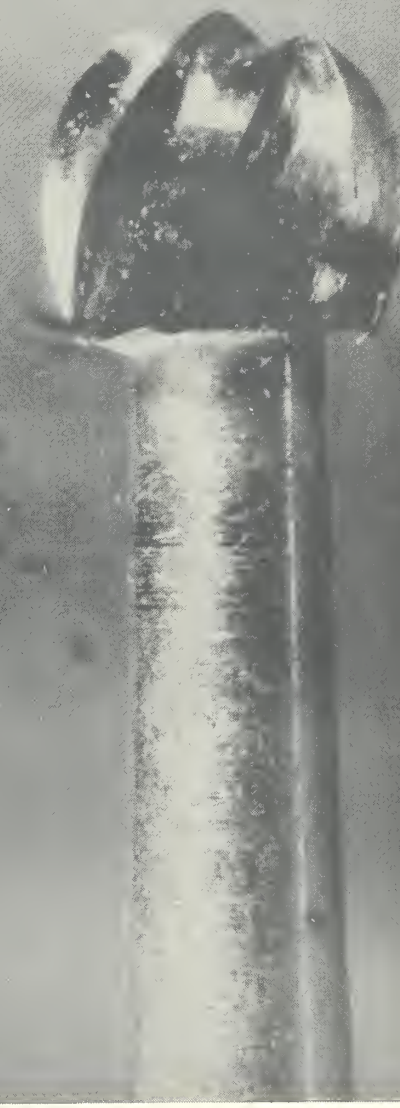
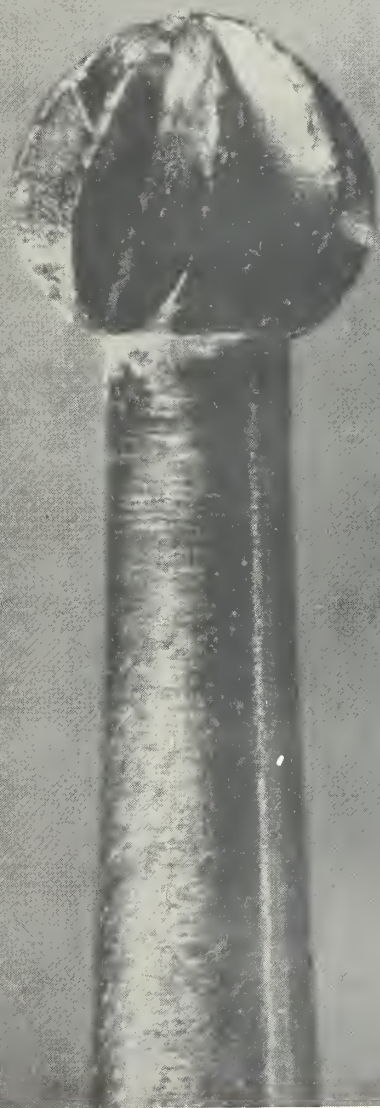


Figure 4

- A. Steel bur after attempting to cut human tooth enamel at 7,500 r.p.m. Note turned edge of blades.
- B. Carbide bur after cutting enamel at 7,500 r.p.m. No apparent damage.

A

B



NBS

Figure 5

Temperature in groove between bur blades, plotted against speed of rotation. Force of 200 grams applied, cutting dentin substitute with #8 steel bur. Shaded areas indicate limits of probable error.

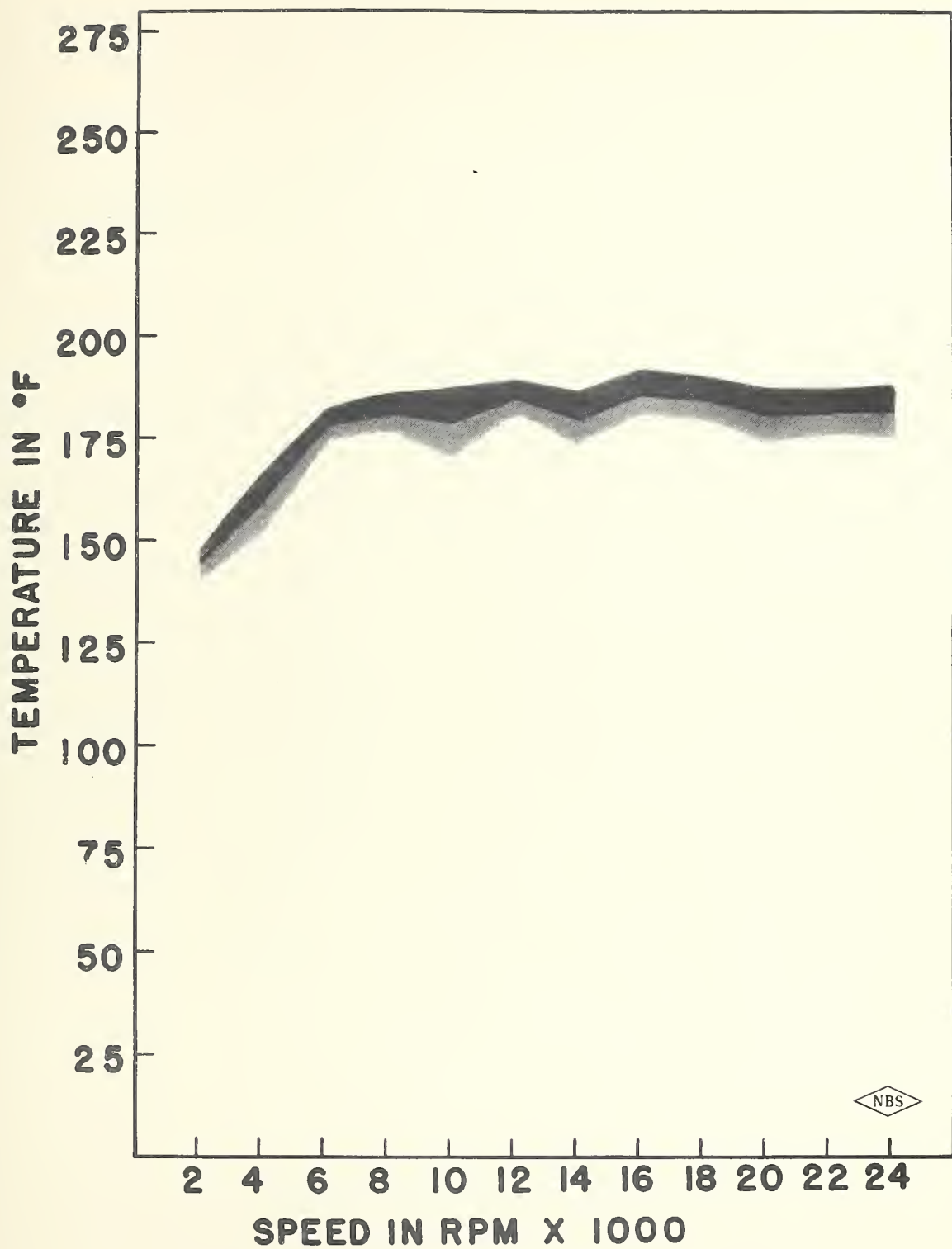


Figure 6

Temperature on slope of bur blade plotted against speed of rotation. Force of 200 grams applied, cutting dentin substitute with #8 steel bur. Shaded areas indicate limits of probable error.

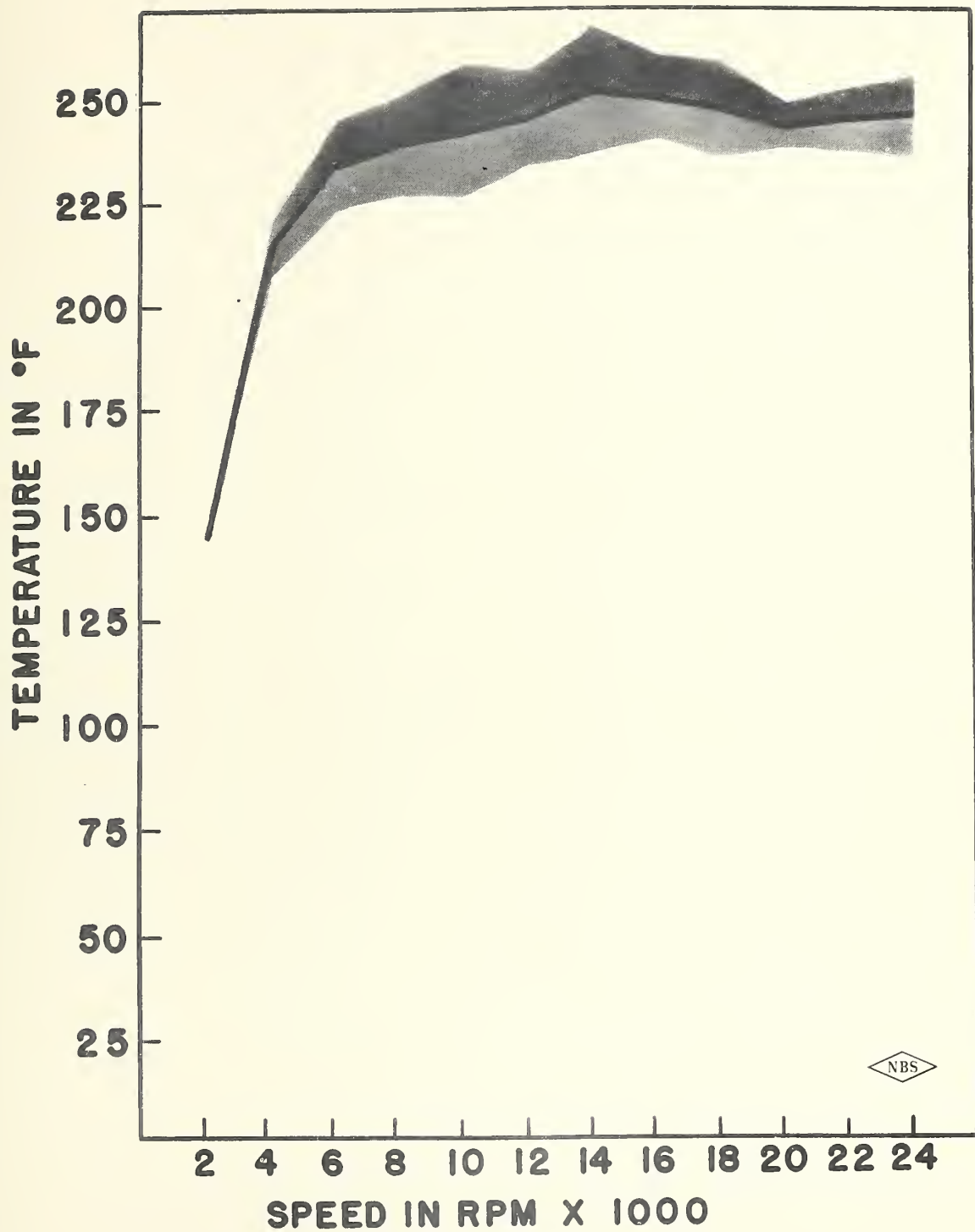


Figure 7

Time required to complete fixed depth of cut to 0.15 inch, plotted against speed of rotation. Force of 200 grams applied, #8 steel bur cutting dentin substitute.

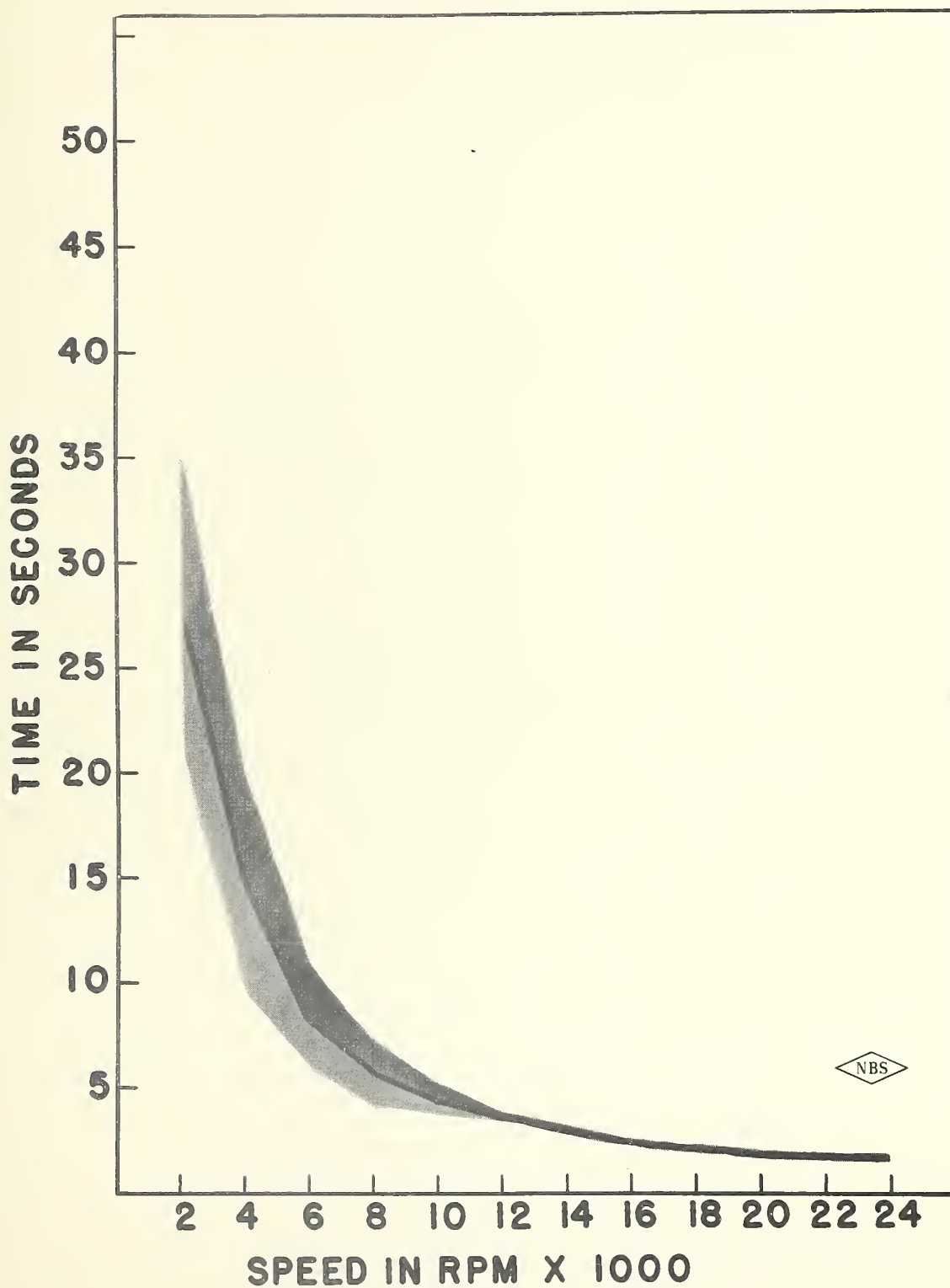


Figure 8

Temperature in groove between bur blades
on #8 carbide bur plotted against speed
of rotation. Force of 200 grams applied,
cutting dentin substitute.

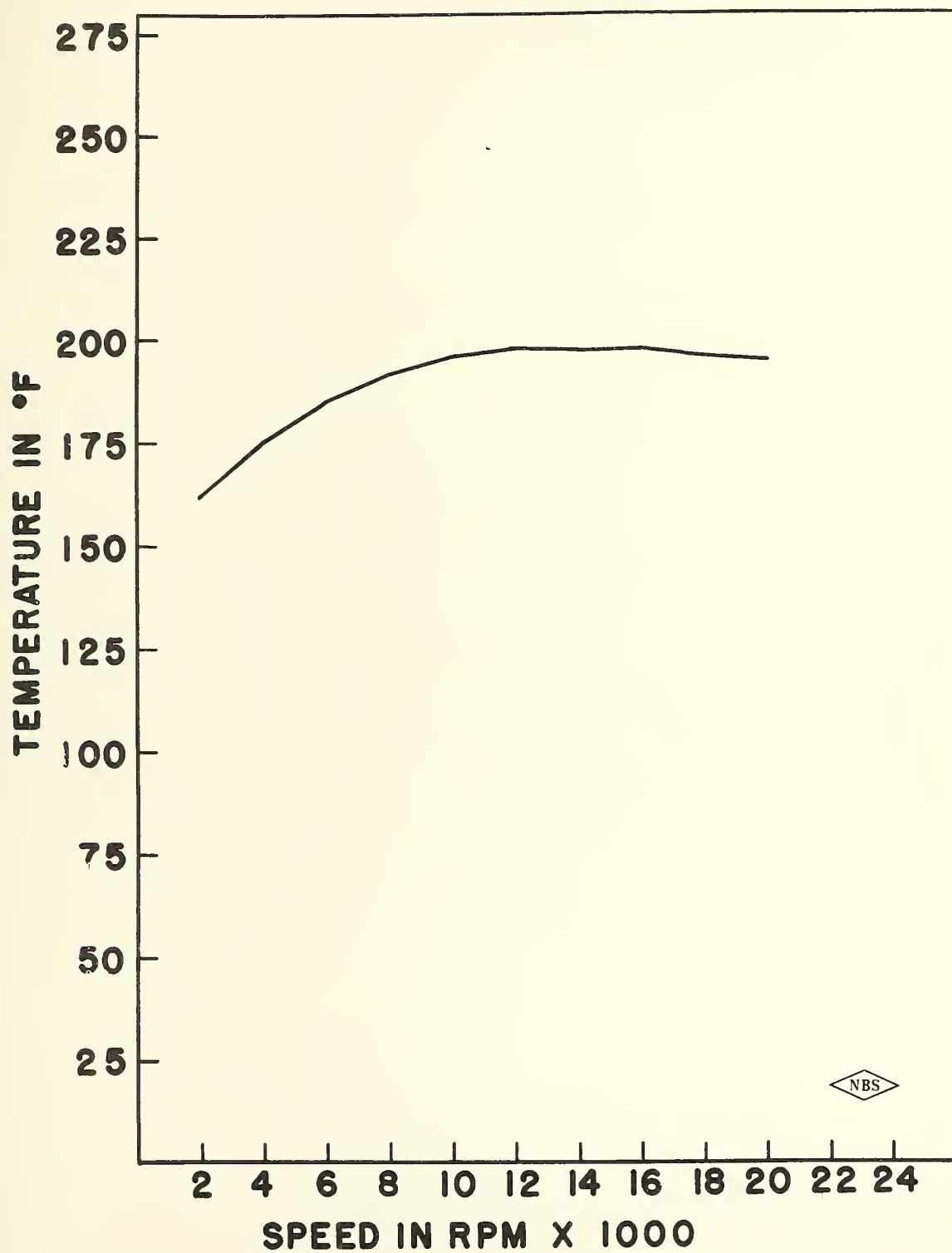
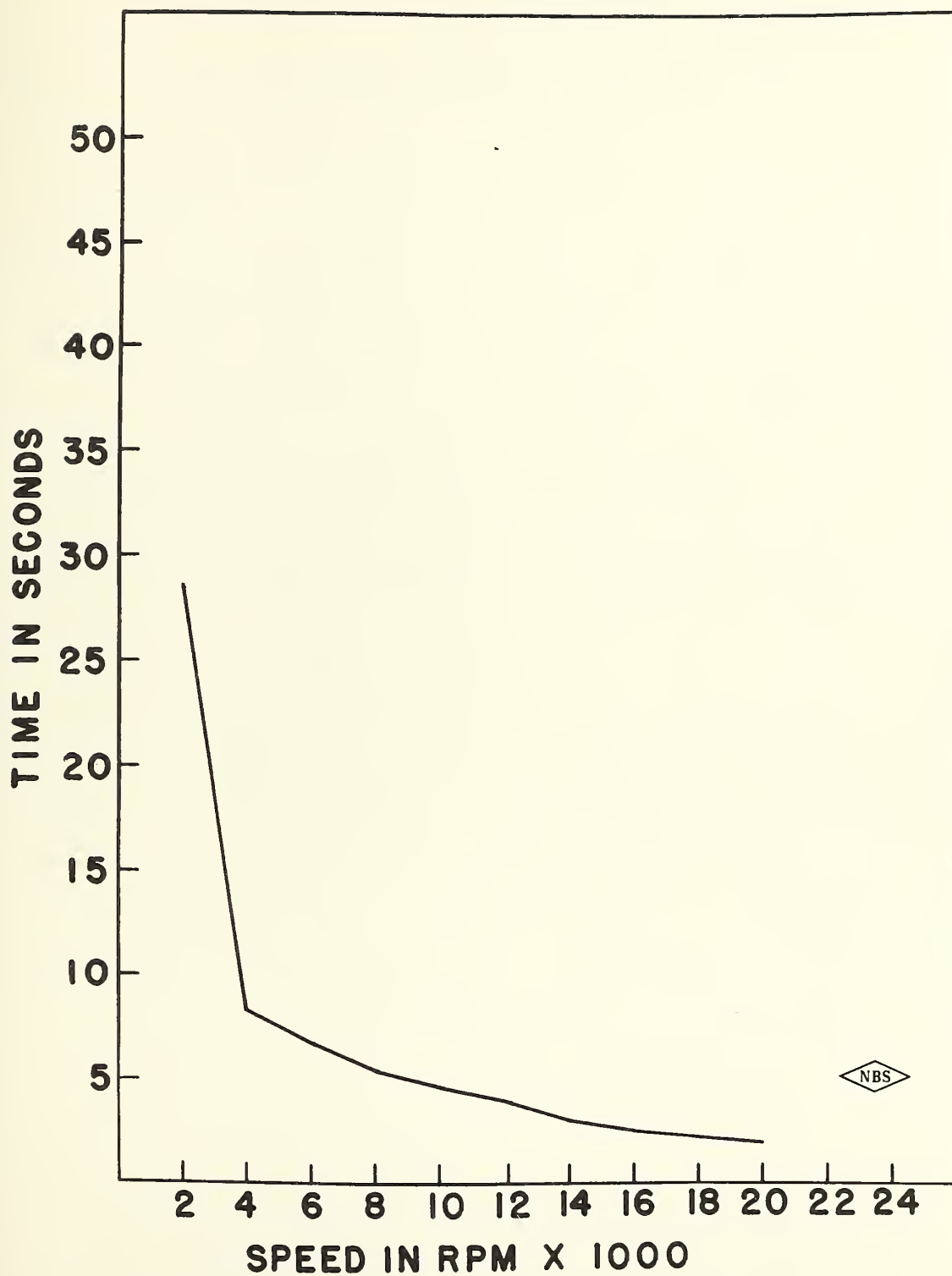


Figure 9

Time required to complete fixed depth of cut to 0.15 inch, plotted against speed of rotation. Force of 200 grams applied to #8 carbide bur, cutting dentin substitute.



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