GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION
SPONSORED PROJECT INITIATION

Date: 7/7/80

Project Title: Effect of Corrosion on Dental Amalgams

Project No: E-19-B05 (continuation of E-19-B04)

Project Director: Dr. Miroslav Marek

Sponsor: DHEW/PHS/ National Institute of Dental Research

Agreement Period: From 7/1/80 Until 6/30/81 (Budget Period)

Type Agreement: Grant No. 5-R01-DE03601-08

Amount: $43,428 PHS
3,094 GIT (E-19-349)
$46,522 TOTAL

Reports Required: Interim Progress Report; Terminal Progress Report

Sponsor Contact Person (s):

Technical Matters

Thomas M. Valega, Ph.D
Chief, Restorative Materials
Program Branch
Extramural Programs, NIDR
Public Health Service
Department of Health, Education, and Welfare
Bethesda, MD 20014
(301) 496-7491

Contractual Matters
(thru OCA)

Robert Ginsburg
Grants Management Officer
Extramural Programs, NIDR
Public Health Service
Dept. of Health, Ed, and Welfare
Bethesada, MD 20014
(301) 496-7437

Defense Priority Rating: None

Assigned to: Chemical Engineering (School/~~Laboratory~~)

COPIES TO:

Project Director
Division Chief (EES)
School/Laboratory Director
Dean/Director—EES
Accounting Office
Procurement Office
Security Coordinator (OCA)
Reports Coordinator (OCA)

Library, Technical Reports Section
EES Information Office
EES Reports & Procedures
Project File (OCA)
Project Code (GTRI)
Other OCA Research Property Coordinator

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION

Date: 7/22/81

Project Title: Effect of Corrosion on Dental Amalgams

Project No: E-19-B05

Project Director: Dr. Miroslav Marek

Sponsor: DHEW/PHS/Nat'l Institute of Dental Research

Effective Termination Date: 6/30/81

Clearance of Accounting Charges: 6/30/81

Grant/Contract Closeout Actions Remaining:

_ Final Invoice and Closing Documents
x ~~Final~~ Fiscal Report (Interim)
x ~~Final~~ Report of Inventions (Interim)
_ Govt. Property Inventory & Related Certificate
_ Classified Material Certificate
_ Other _____

Continued by E-19-B06

Assigned to: Chemical Engineering (School/~~Laboratory~~)

COPIES TO:

Administrative Coordinator
Research Property Management
Accounting Office
Procurement Office
Research Security Services
~~Reports~~ Coordinator (OCA)
Legal Services (OCA)
Library, Technical Reports
EES Research Public Relations (2)
Project File (OCA)
Other: Suspense - 9/30/81

GEORGIA INSTITUTE OF TECHNOLOGY
ATLANTA, GEORGIA 30332

OFFICE OF
THE
COMPTROLLER

October 16, 1980

Grants Management Officer
National Institute of Dental
Research
Public Health Service
DHHS
Bethesda, Maryland 20205

Dear Sir or Madam:

Enclosed is the Report of Research Grant Expenditures for Grant No. 2 R01 DE03601 -07 covering the period 7/1/79 - 6/30/80.

If you have questions or require additional information, please let us know.

Sincerely,

David V. Welch, Manager
Grants and Contracts Accounting

DVW/BITS/jb
Enclosure
cc: Dr. M. Marek
Dr. G. W. Poehlein
Mr. H. Dean
Mr. O. H. Rodgers
File E-19-B04

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AN EQUAL EDUCATION AND EMPLOYMENT OPPORTUNITY INSTITUTION

DEPARTMENT OF HEALTH AND HUMAN SERVICES

(Instructions are on reverse)

NAME AND ADDRESS OF GRANTEE INSTITUTION	TRANSACTION NO.	Grant No. 2 R01 DE03601-07
Georgia Institute of Technology Atlanta, Georgia 30332	(08)RIDE03601C	DATE OF THIS REPORTING PERIOD FROM 7/1/79 TO 6/30/80
	INSTITUTIONAL ID NO. E-19-B04	PROJECT PERIOD FROM 6/1/76 TO 6/30/83
		☐ CHECK IF FINAL REPORT

1. Expenditures of DHHS Funds for this Reporting Period

a. Personnel	$	h. Alterations and renovations	
b. Consultant services		i. Other	
c. Equipment			
d. Supplies		j. Total direct costs	$ 25,849.90
e. Travel, domestic		k. Indirect costs: Rate 76 % ☒ S&W ☐ TDC	
f. Travel, foreign		Base $ 21,025.49	15,979.37
g. Patient care costs		l. TOTAL	$ 41,829.27

2. Expenditures from Prior Periods (previously reported)	-0-
3. Cumulative Expenditures	41,829.27
4. Total Amount Awarded – Cumulatively	41,844.00
5. Unexpended Balance (Item 4 less Item 3)	14,73
6. Unliquidated Obligations	-0-
7. Unobligated Balance (Item 5 less Item 6)	14.73
8.a. Cost Sharing Information – Grantee Contribution This Period	4,992.94
b. % of Total Project Costs (Item 8a divided by total of Items 1 and 8a)	% 10.7
9.a. Interest/Income (enclose check)	-0-
b. Other Refundable Income (enclose check)	-0-

10. Remarks

I hereby certify that this report is true and correct to the best of my knowledge, and that all expenditures reported herein have been made in accordance with appropriate grant policies and for the purposes set forth in the application and award documents.

Dr. M. Marek — Associate Professor — Date

SIGNATURE OF INSTITUTION OFFICER — DATE 10/16/80

David V. Welch, Manager, Grants & Contracts Acctg.

Formerly HE.-469 404/894-4624

REPORT OF RESEARCH GRANT EXPENDITURES

E-19-B05

SECTION IV

APPLICANT: REPEAT GRANT NUMBER SHOWN ON PAGE 1	GRANT NUMBER	
SECTION IV—SUMMARY PROGRESS REPORT	DE 03601-09	
PRINCIPAL INVESTIGATOR OR PROGRAM DIRECTOR (Last, First, Initial) Marek, Miroslav	PERIOD COVERED BY THIS REPORT FROM	THROUGH
NAME OF ORGANIZATION Georgia Institute of Technology	06/01/81	06/30/82

TITLE (Repeat title shown in Item 1 on first page)
EFFECT OF CORROSION ON DENTAL AMALGAMS

1. List all publications, not previously reported, resulting from work supported by this grant (author(s), title, page numbers, year, journal or book). List manuscripts separately as submitted for publication or accepted for publication.
2. Provide two reprints of publications not previously submitted to the awarding unit.
3. Progress Report. (See instructions)

Publications

M. Marek: "Corrosion of Dental Materials," in Treatise on Materials Science and Technology (J.C. Scully and J. Castle, eds.), Academic Press (in press).
M. Marek: "Corrosion of Dental Materials," in Encyklopedia of Materials Science and Engineering (M.B. Bever, ed.), Pergamon Press (in press).

Conference Presentation:
P. Biltoft, R. F. Hochman, and M. Marek: "The Effect of Porosity on Corrosion of Dental Amalgam." IADR/AADR Annual Meeting, March 19-22, 1981.

Progress Report

1. OBJECTIVES

A. Overall Objectives

To improve the understanding of the role of corrosion in the deterioration of dental amalgam restorations by investigating the mechanism of corrosion, the effects of corrosion on the properties, and by developing suitable testing methods.

B. Goals for the Current Year

a. To examine the breakdown of the Cu_6Sn_5 phase which is involved in the mechanism of corrosion of high-copper dental amalgams.
b. To examine the relationship between mechanical properties and corrosion.
c. To evaluate the effect of porosity on corrosion of dental amalgam.
d. To evaluate the synthetic electrolytes used in the corrosion tests.

2. STUDIES AND RESULTS

a. A Study of the Mechanism of Corrosion of High-Copper Dental Amalgam: Breakdown of the Cu_6Sn_5 Phase

Samples of the η' (Cu_6Sn_5) phase were prepared by melting pure components in vacuum. Some of the samples contained high percentages of a tin-rich phase; the most homogeneous samples were used in the tests. The composition and structure were verified by microprobe and X-ray analyses.

The corrosion properties of the η' phase were determined by measuring the potentiodynamic anodic polarization behavior, corrosion potentials, and potential differences between the η' phase and other phases of the amalgam microstructure. All tests were made in the following electrolytes: synthetic saliva, 1% NaCl (neutral), and 1% NaCl acidified to pH 1. The acidified solution was used to simulate conditions in occluded corrosion cells.

The results can be summarized as follows: in nearly neutral synthetic saliva the η' phase suffers little deterioration; the anodic polarization curves show no breakdown of passivity, and the corrosion potential differences between the phase and the γ and γ_1 phases were within 0.1 V. In 1% NaCl the activity of the η' phase increased but the corrosion rate remained relatively low in the range of potentials of interest. The highest activity was observed in the acidified

solution; however, even in this case, the corrosion resistance was close to that of the other phases.

The results indicate that deterioration of the η' phase in dental amalgam cannot be explained on the basis of the susceptibility of Cu_6Sn_5 alone. The possible explanations involve effects of impurities, differences from stoichiometry, and the effect of the presence of other phases. These relationships will be examined on special samples in which these parameters have been varied.

b. Relationship Between Mechanical Properties and Corrosion

A study has been initiated on the effect of creep deformation on the corrosion behavior. In the first part, which has been completed, ADA specimens were exposed to synthetic saliva or 1% NaCl at 37°C and subjected to a compressive load of 36 MPa. After one hour of loading the following corrosion tests were performed: anodic polarization measurement, and coulometric test at constant potential of 0.0V (SCE). Free corrosion potential was also measured for the entire duration of the creep test. For all creep-corrosion measurements control tests were performed under the same conditions except without the application of the load.

The results have shown that compressive creep deformation has very little effect on the corrosion behavior. At the application of the load the corrosion potential changed to a value more negative by about 10 mV; this change, however, decreased to zero within a few minutes. The polarization curves and coulometric data were virtually unaffected by the load.

In view of the reported effects of tensile creep on corrosion it has to be concluded that only tensile deformation has a substantial effect. A constant load pneumatic tensile testing machine has been adapted for creep tests of amalgam specimens, and preliminary tests were made. The detailed study will be performed during the next phase of the program.

c. The Effect of Porosity on Corrosion of Dental Amalgam

A thorough study of the porosity and its effect on the corrosion behavior was performed. The study was made on a high-copper, single composition alloy which shows relatively high corrosion activity. The samples were prepared at different initial Hg/alloy ratio but at nearly constant final Hg content. The porosity was evaluated by metallography, gas adsorption analysis, and vacuum impregnation with liquids. The evaluation included determination of number of pores in various size classes, determination of the form factor, and calculation of the true surface area, which included the surface area of the open pores. Corrosion tests were made in 1% NaCl by the coulometric constant-potential method. Tests were also made on samples impregnated with epoxy resin which filled the pores.

The results showed that the porosity increased with decreasing Hg/alloy ratio. The true surface area of a specimen of a nominal 1 cm^2 area increased from 2.99 cm^2 for 54% Hg to 3.99 cm^2 at 50% Hg to 6.64 cm^2 for 46% Hg (initial mercury contents). The corrosion index, obtained from the coulometric tests, increased linearly with the true surface area. Tests with epoxy-impregnated specimens showed lower corrosion index and corrosion depth after epoxy-impregnation. The study is continuned to examine if there is association of some of the phases with pores in the high copper amalgams, and to evaluate the significance of the occluded cell formation in the pores.

d. Evaluation of the Synthetic Electrolytes

Seven synthetic electrolytes, which have been reported in the literature as substitutes for the oral environment in corrosion tests, have been examined. The Redox potential, pH, and buffering capacity were measured for each electrolyte. Titration curves were also obtained for each electrolyte. The results show that, generally, only synthetic salivas which contain bicarbonates and CO_2 approximate human saliva in the buffering capacity, which is important in the corrosion processes.

3. SIGNIFICANCE

The proposed research will contribute to the understanding of the basic processes of deterioration of dental amalgam restorations and thus help in the development of improved materials.

4. RESEARCH GOALS FOR THE COMING YEAR

a. A Study of the Mechanism of Corrosion of High Copper Amalgam: The breakdown of the η' phase will be further examined using specimens of different purity and poly-electrodes in which the phase is surrounded by other components of the microstructure.

b. A Study of the Effects of Aging on the Corrosion of High Copper Amalgam: Test have shown that the corrosion resistance of high copper amalgam increases with time even for amalgams in which no γ_2 has been detected. A study of this phenomenon may contribute to the understanding of the corrosion mechanism.

c. A Study of the Effect of Tensile Creep Deformation on the Corrosion Behavior: A continuation of the present study will be focused on tensile creep and the relationship of the corrosion and creep effects with the breakdown of margins of restorations.

d. A Study of the Effect of Porosity on Corrosion of Dental Amalgam: In a continuation of the present study the mechanism of corrosion in the pores will be examined.

The undersigned agrees to accept responsibility for the scientific and technical conduct of the project and for provision of required progress reports if a grant is awarded as the result of this application.

4-21-81
Date

Principal Investigator