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AN INVESTIGATION OF METAL TO METAL UNIONS
USED IN DENTISTRY WITH SPECIAL REFERENCE
TO ELECTRODEPOSITION PROCEDURES.

A Thesis submitted to the University of Sydney
in support of my candidature for the degree of
Doctor of Dental Science.

Olbert William Rogers, B.D.S.

November, 1972.

The University of Sydney

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The work presented in this thesis has been carried out by me as an individual research project and I have personally prepared and examined all of the specimens used in this survey.

The electroplating facilities at the Sydney Technical College were made available to me by Mr. B. Armstrong, Lecturer, School of Metallurgy, University of New South Wales, for all the research that involved electro-deposition of metals.

To develop a suitable electrolyte for electroforming a gold matrix I am further indebted to Mr. Armstrong for his technical assistance and supervision. Consequently this initial project was published conjointly.

The friendly help and encouragement of Mr. A. Docking and Mr. J. Griffith of the Commonwealth Bureau of Dental Standards and their willingness to provide the many facilities available at the Bureau, made the years of research involved a pleasant experience.

I am most appreciative of having been able to participate in and utilise the excellent liaison that exists between the Faculty of Dentistry and its allied scientific fields.

This thesis was previously presented in November 1972 and, as requested by the Board of Postgraduate Studies, has been revised for resubmission.

The thesis is basically unchanged except for the provision of statistical data, clarification of tables, additional control specimens, and a reassessment of the objects of investigation, and the summary and conclusions to Parts 11 and 111. An introduction to describe the theme of the published work and how the various publications are related to one another and the theme has also been included.

I would like to express my indebtedness to Associate Professor G. Wing, Acting Head of the Department of Operative Dentistry, Faculty of Dentistry, The University of Sydney, for his assistance in the revision of the thesis.



Olbert William Rogers, B.D.S.

October, 1973.

The initial research program was to develop an indirect gold inlay technique that permitted marginal adaptation of the inlay to the tooth surface and thus provide compensation for the series of inaccuracies that occur due to the properties of the various materials used in the production of the gold inlay. Further, the use of harder gold alloys for inlays is limited by their poor response to burnishing and spinning procedures used to improve marginal adaptation.

Thus the concept, of casting into a pre-formed pure gold matrix to provide an inlay with margins that are independent of the type of gold alloy selected for casting, was investigated.

The complexity and individuality of cavity design predicated electroforming as the only satisfactory means of producing such gold matrices. Three initial problems existed.

1. No suitable electrolyte was available for heavy electrodeposition of gold.
2. A procedure to make the surface of artificial stone electrically conductive to initiate the electrodeposition of gold had to be found as the conductive agents used for the electroforming of dental dies (i.e. colloidal graphite, silver flake and precipitated silver) were unsatisfactory.
3. Casting to embedded gold was discouraged in the literature and the soldering of the interface between such unions after casting was recommended. The requirements of the matrix inlay procedure depended on the electroformed gold matrix and the cast gold alloy

becoming an integral unit that would not separate under function or manipulation and the union should be such that it would not permit penetration of saliva or bacteria. Consequently the type of union at the interface had to be established and its efficacy determined.

To develop the gold electrolyte, facilities and assistance were afforded by Mr. B.W. Armstrong through Prof. R. Myers at the University of New South Wales. The consequent results of this investigation were published conjointly along with a general outline of the indirect inlay procedure and its feasibility as a dental procedure.

Rogers, O.W. and Armstrong, B.W. - Electroforming a gold matrix for indirect inlays. J. Pros. Den., 11:5, 955-966 (Sept. -Oct.) 1961.

The use of electrodeposition procedures, particularly that of electroforming, has been neglected by the dental profession. Apart from the production of silver or copper electroformed dies and a limited use of electroplated gold or rhodium for aesthetics or protection of the underlying basis metal, the dentist has depended upon casting or manipulative procedures such as swaging, rolling or bending to produce metal appliances.

The realisation of this indifference or oversight to exploit applications of electrodeposition procedures resulted in the following publications.

Rogers, O.W. - Electrodeposited copper individual impression trays. J. Pros. Den., 16:1, (Jan. -Feb.) 1966.

Rogers, O.W. - Gold plating of cobalt-chromium. Austral. D.J., 14:2, 79-81, (Apr.) 1969.

Rogers, O.W. - Electroformed metal palates for complete dentures. J. Pros. Den., 23:2, 207-217, (Feb.) 1970.

The initial electroformed gold matrix construction was then expanded into a detailed indirect inlay technique with consideration to its clinical application.

Rogers, O.W. - The electroformed gold matrix inlay technique. Austral. D.J., 15:4, 316-326, (Aug.) 1970.

In order to evaluate the effectiveness of the union between the electro-plated metals and the basis metals a metallographic investigation, as in the body of the thesis, was commenced.

The study was then expanded to include other unions used in dentistry and was carried out in two parts;

1. Unions in precious metal alloys.
2. Unions in chromium containing alloys.

Because of the lack of information concerning these unions the use of metallographic methods was decided upon and techniques were developed in order to investigate the unions of dissimilar metals. This investigation was further extended to examine and to effect a method of preventing crevice corrosion in silver soldered stainless steel joints.

All these procedures involving electrodeposition, electrolytic polishing and etching, and metallic corrosion are aspects of the same phenomenon. Stated simply, they all involve the transfer of metallic

ions through an energy barrier that exists at the interface between a metal and an electrolyte.

For convenience, the thesis is divided into three sections, each having a section on materials and methods, results, consideration of results, and a summary and conclusions. A general conclusion is then made on the entire body of the thesis.

Please note.

The size of the photomicrographs has been reduced approximately twenty five percent in this copy of the thesis with the exception of figures 40, 50A and 90B.

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INTRODUCTION

Just as man's progress has been dependent upon the discovery, production and employment of metals, the progress of dentistry has also been largely influenced by the use of suitable metals. Indeed there are very few facets of dentistry in which metals have not assumed a paramount place.

A high degree of individuality is required in the fabrication of dental restorations and appliances and in many instances the induced physical properties of the materials must be maintained. It is physically as well as economically impossible to fabricate these restorations and appliances by any other method than that of joining. Not only is the union of metals used to fabricate restorations and appliances but in a broader concept metal unions may be employed for protection from corrosive conditions or for aesthetics e.g. electroplating.

Examples of metal to metal union used in dentistry are:

A. Restorative dentistry.

1. Inlay gold cast against a pure gold matrix
2. Gold alloys cast onto preformed backings
3. Fabrication of bridges
4. Contact point adjustment of gold inlays
5. Repair to bridges and crowns
6. Electroplating of dissimilar golds.

SUMMARY AND CONCLUSIONS.

PART 1. PRECIOUS METAL ALLOYS.

B. Orthodontics.

1. Fabrication of bands
2. Attachment of brackets and tubes to bands
3. Joining of wires and attachment of hooks, stops and springs
4. Electroplating of stainless steel appliances.

C. Prosthetics.

1. Attachment of clasps
2. Production of individual impression trays
3. Electroplating of cobalt-chromium and electroformed dental palates
4. Repair of clasps and metal dentures.

D. Oral surgery.

1. Fabrication of splints.

E. Periodontics.

1. Fabrication of splints.

All of these metal unions are accomplished by either soldering, welding, casting against or electrodeposition. Ideally the requirements for soldered unions are:

- a. complete wetting
- b. strength
- c. flow
- d. bonding
- e. freedom from pitting

- f. retention of inherent physical properties
- g. resistance to corrosion.

These requirements would also apply to welded unions and cast unions. Electrodeposited unions need to be continuous, adherent and protective.

Problems associated with the joints and appliances described.

Failure of jointed restorations or appliances in all cases is due to loss of strength in the basis metal or joint, fracture of the basis metal, the joint of the joining metal, or the corrosion of the joint.

Because of the corrosive nature of the oral environment there is a limitation to the type of metals used in dentistry and therefore noble metals with their high resistance to corrosion were the first materials used. Later the development of corrosion resistant base metals enabled dentists to take advantage of the lower density and the lower cost of these materials. Although in most cases the corrosion resistance of these base metals approaches that of the noble metals, failures due to corrosion still occur.

REVIEW OF THE LITERATURE.

REVIEW OF THE LITERATURE.

Part 1.

Precious Metal Alloys.

The technique of gold soldering for the making of dental appliances has been known to man for 25-27 centuries. Peyton¹ states that "the practice of using gold crowns for bridgework apparently flourished in Etruria and Rome as early as 700-500B.C. The arts of soldering and riveting must have been well understood in order to prepare a restoration from pure gold rings soldered in correct relations, with the artificial tooth held in place by a pin which passed through the artificial tooth and the gold ring."

Tylman² describes an Etruscan dental bridge constructed before 500B.C. "it was made of a series of seven light gold rings soldered together, five of which embraced upper natural teeth, one holding an artificial left second bi-cuspid, the other retaining a block of two artificial central incisors riveted in place, and it probably was carved from a single tooth of an ox or a calf."

The soldering of these appliances indicates a knowledge of gold alloys and the use of fluxes and perhaps anti-fluxes.

There were no improvements over the early Etruscan appliances until the early eighteenth century when Pierre Fauchard published his work *Le Chirurgien Dentiste*.

An additional application for dental soldering occurred with the introduction of the matrix inlay. According to McGehee, True and Inskipp³ in 1837 John Murphy of London produced the original platinum-foil matrix for porcelain inlays and the first gold inlays. The matrix was adapted to the cavity in the tooth, removed and filled with gold solder. The inlay was then polished and cemented back into the tooth.

From the single matrix technique the double matrix technique evolved. One gold matrix was formed to fit into the cavity and a second matrix was contoured to form the lost external surface of the cavity. The space between the matrices was filled with the gold solder. The full development of this soldering technique can be seen in the papers of Hinman⁴ (1906).

In 1907 Taggart evolved the cast gold inlay⁵ and the gold matrix technique was superseded. However gold soldering assumed greater importance in the joining of cast bridge units,^{6,7} the contouring of contact points⁸ and the repairing of gold appliances.⁶

Wrought gold alloy wires and bands were extensively used for orthodontic bands and required soldering,⁹ and swaged gold palates for full dentures had retentive attachments soldered to them.¹⁰ Gold alloy wires, clasps or cast gold alloy clasps are still soldered to the framework of partial dentures.¹¹

Thus gold solders are used for the joining of gold alloys that are either in the cast or the wrought condition.

Another type of gold to gold union occurs when a gold alloy is cast to another gold alloy. Ney (1958)¹² stated that "even with modern casting technics it is virtually impossible to cause a complex casting gold alloy to adhere perfectly to a similar gold alloy by the 'casting-to' technic. Regardless of the excellent appearance of a 'cast-to' joint it is advisable to join the parts together with a fine line of solder."

In 1961 Rogers and Armstrong¹³ published an inlay technique that required a dental gold alloy to be cast to an electroformed gold matrix. Rogers¹⁴ published further details of this procedure in 1970.

Peyton et al. (1964)¹⁵ reported good union between cast gold alloy and clasp wire and Asgar and Peyton,¹⁶ and Gibson, Asgar and Peyton¹⁷ had publications in 1965 showing union between cast gold alloys and embedded metals.

The Jelenko Company (1965)¹⁸ stated "when casting gold to gold - whether it be a casting to a partial denture, bridge, or coping - the joining of metals is somewhat similar to soldering."

Undoubtedly, "casting-to" techniques are now established and this type of union will be further evaluated in a following section.

Ante (1918)¹⁹ considered that "in its application to dentistry soldering has assumed the significance of an art of much greater importance than ever attained by the gold or silversmith," and listed the essential requirements for successful soldering as:

1. Cleanliness of the surface
2. Proper flux
3. Apposition of surfaces
4. A free flowing solder
5. Proper amount and distribution of heat
6. Proper support.

Coleman(1928)^{20,21} published the composition, melting range and physical properties of five gold solders. Study of the microstructure of the soldered joint showed recrystallisation of wrought dental gold wires when overheated during the soldering procedure. An ideal strong soldered joint was defined as one that shows no solution of the wrought wire and he observed that grains at the surface of the soldered joint may act as nuclei for grains forming in the solidifying solder.

Taylor and Teamer(1949)²² recommended fusion tests for assessing the joining properties of gold solder. They also listed the desirable physical characteristics of a solder as:

1. Easy flowing
2. Free flowing
3. Freedom from pitting
4. Strength approximately or exceeding that of the underlying structure

5. Colour to match the materials with which they are used
6. Building qualities which permit the development of desired shapes by partial fusion
7. Freedom from corrosion or staining in service.

Ryge (1958)²³ also established by metallographic examination that a strong junction can be obtained without any noticeable diffusion between the solder and the parent alloy. At increased temperatures penetration along the grain boundaries of the parent metal occurred with eventual diffusion and alloying between the solder and the parent metal.

O'Brien, Hirthe and Ryge (1963)²⁴ investigated the wetting characteristics of two dental solders by the droplet method. This is a static technique and is dependent on measuring the contact angle formed between the solder and the basis metal. Procedures based on the measurement of the contact angle have received criticism^{25,26,27} as infinitesimal impurities, technical stresses, surface conditions and temperature all have an influence upon the contact angle.

Alternative techniques for studying the surface tension of metals and alloys are highly complex and their advantages and disadvantages are discussed by Semenchenko.²⁸

Anderson²⁹ relates solderability to the contact angle and the pioneering work of O'Brien et al.²⁴ to produce a qualitative procedure

to evaluate dental soldering procedures will undoubtedly influence future research.

El-Ebrashi, Asgar and Bigelow(1968)³⁰ examined gold soldered joints with the electron microscope. They confirmed that strong soldered joints can be obtained without diffusion occurring between the solder and the parent alloy. Pronounced oxide formation, microporosity and alloying with the parent alloy were observed in overhead joints.

The laser welding of gold alloys was investigated by Smith, Burnett and Gordon³¹ (1972). They found that the strength of laser welded joints was more uniform than solder joints of the same alloy, but the hardness and the tensile strength values were comparable.

Part 11.a.

Stainless Steel.

As is true for many scientific discoveries, no single nation can claim full credit for the development of stainless steel.

According to Gaston³²; Brearley of England, Strauss and Maurer of Germany and Haynes of America quite independently developed corrosion resistant steel.(1911-1914)

Austenitic stainless steel in the form of hard drawn wires was introduced to the dental profession in 1929³².

To develop the full potential of the properties and advantages of stainless steel a technique of joining had to be developed, as the solders and fluxes used in the fabrication of gold appliances were ineffective with stainless steel.

Anderson(1933)³³, Golden(1934)³⁴ and Brusse and Carmen(1934)³⁵ advocated the use of fluoride fluxes. To assist in the production of stainless steel joints gold solders were mainly used, but their high fusion range caused recrystallisation of the stainless steel with the consequent loss of the physical properties. Freil(1934)³⁶ suggested the use of the lower fusing silver solders.

Although Kadesky(1935)³⁷ reported no difficulty in soldering stainless steel appliances, the majority of operators - Freil(1934)³⁶, Mogler and Holestine(1935)³⁸, Yost(1936)³⁹, Walsh(1938)⁴⁰ and Carmen

(1938)⁴¹ - considered soldering as impractical due to the high failure rate of soldered joints, softening of stainless steel after soldering, and the difficulty in the soldering procedure. They all recommended spot welding techniques, as developed by Walsh⁴⁰, and Brusse and Carmen in 1933³⁵.

In 1935 Souder⁴² stated "attempts at welding or soldering these base metal alloys have presented considerable difficulty. No great amount of time has been spent at the Bureau* on these base metal alloys used in dentistry since they do not appear to possess any superior physical properties and do possess some definitely inferior properties."

However, the momentum of research on stainless steel was maintained. Its exceptionally high tensile strength, hardness, corrosion resistance and lower density offered lighter and stronger appliances for the orthodontist.

Golden(1934)³⁴, Freil(1934)³⁶ and Yost(1936)³⁹ found that the oral tissues showed greater tolerance to stainless steel than the gold alloys, and there was greater freedom from the adherence of plaque and debris. Yost stated "I do not in any sense consider chrome alloys as a substitute for gold alloys, but an evolutionary product of progress."

Vosmick and Taylor(1936)⁴³ soldered stabilised stainless steel with 14K and 10K gold solders and a silver solder. They found that

* National Bureau of Standards, Washington, D.C.

the tensile strength of the silver solder was in excess of that of the gold solders and the greater the gold content of the solder the weaker the joint. Photomicrographs showed no superficial alloying of solder and metal, there being a sharp line of demarcation where the solder and alloy came into contact with each other. From their tensile strength measurements they concluded that the strength of the joint was dependent on the adherence of the solder to the metal. Prior to this the joint was considered to be purely mechanical in nature. They also established that transformation would occur in the stainless steel at temperatures in excess of 720°C - 750°C after heating for 15 seconds.

Greenwood (1937)⁴⁴ noted the loss of corrosion resistance to stainless steel that had been heated to a temperature of 500°C - 700°C and Down (1937)⁴⁴ and Worner (1937)⁴⁴ reported that casting gold to stainless steel posts led to carbide precipitation.

Wunderly (1945)⁴⁵ and 1960)⁴⁶ presented a technique for soldering with the flame from a hypodermic needle and considered the joint mechanical in nature.

Chatfield and Swift (1949)⁴⁷ published details on stress cracking corrosion, and corrosion due to the presence of chloride ions in silver soldered, stainless steel joints.

Coxe and Setapen (1949)⁴⁸ advanced the "triaxial tension" theory

to explain how silver brazing alloys with a tensile strength of 70,000 lbf./in.² can withstand a stress of 130,000 lbf./in.²

Case (1950)^{49,50,51} and Henns (1951)⁵² studied the stainless steel, silver solder joint interface and concluded that interalloying* did occur whilst Richman (1956)⁵³ and Bien and Ayers (1959)⁵⁴ contended that the joint was mechanical in nature. Bien and Ayers (1959)⁵⁵ also published results showing the presence of fluoride flux at the joint interface and in 1958⁵⁶ considered the effect of soldering on electrodeposited stainless steel to reduce the incidence of crevice corrosion.

Brooker and Beatson (1953)⁵⁷ suggested the addition of borofluoride and fluoborate fluxes to borax based fluxes to provide a flux that melts below 600°C yet retains its full effectiveness up to 750°C.

Wilkinson (1960-1963) showed that the hardness of stainless steel wire was directly proportional to its tensile strength.⁵⁸ Thus he was able to calculate the effect of soldering upon the proportional limit of stainless steel by the systematic hardness testing of the stainless steel in the joint area, and so was able to compare different soldering techniques.⁵⁹

He established that the optimum soldering temperature for low fusing solders (M.P. 615°C) was 20°C to 40°C above the flow point of the solder and was able to determine that excessive softening of stainless steel wire is related more to higher temperatures than longer

* The term interalloying is used to describe diffusion across the interface of two alloys in contact as distinct from alloying in the metallurgical sense where two alloys or metals are melted together.

heating time.⁶⁰

In 1964 the Australian Standard (T30-1964) for silver solder was introduced.

Gardiner and Aamodt (1969)⁶¹ re-evaluated silver soldering procedures, and from their metallographic examination and tensile test results they concluded that the union was more than mechanical and interalloying possibly occurred.

Adams (1970)⁶² received a research grant to study fusion welding of dental alloys with a pulsed ruby laser.

Williams and Von Fraunhofer (1971)⁶³ published an interim report on an electrochemical means of studying stainless steel for "the selected dissolution or etching of different structural phases," of stainless steel orthodontic wires to correlate microstructure with the composition and physical properties of the wire.

Laird and Von Fraunhofer (1972)⁶⁴ compared an electro brazing technique and gas-air brazing of silver solder joints on stainless steel. No significant difference occurred between the soldered joints examined and an electromicroprobe analysis did not reveal any trace of inter-metallic compounds at the solder, steel interface.

One major point is evident throughout the literature on the silver soldering of stainless steel - there is divergence of opinion on whether the joint formed is mechanical or whether interalloying occurs.

Due to the passivity of stainless steel, etching procedures required to bring up its structure lead to a loss of levelling at the interface due to preferential dissolution of the silver solder. Thus results have had to be based on presumption rather than direct observation owing to the metallographic difficulties encountered.

Part 11, b.

Cobalt-chromium alloys.

The alloys of cobalt and chromium were first introduced to the dental profession in 1929.⁶⁵ According to Lane, cobalt-chromium dentures have been worn since 1931 and in 1932 the alloy was patented.⁶⁶

Because of the high casting temperature, and the hardness of this material, special equipment is required for its fabrication. As a result the production of cobalt-chromium dental appliances has become restricted to specialist dental laboratories.

According to Brockhurst (1971),⁶⁷ the first Australian cobalt-chromium denture was produced in 1932 and the first Australian laboratory was established in 1936.

Dental literature on cobalt-chromium alloys has largely been concerned with its physical properties, structure and composition.^{68,69,70,71,72} There is very little information available about soldering procedures because the repairing, as well as the manufacture of these appliances, is carried out in commercial dental laboratories.

The joining techniques that apply to stainless steel can also be applied to the cobalt-chromium alloys because cobalt-chromium and stainless steel are similarly dependent upon their chromium content for passivation.⁷³

Anderson⁷⁴ states that the cobalt-chromium alloys can be soldered with 18K gold solder, the parts being invested and soldered in a furnace containing a reducing gas such as hydrogen. Some commercial laboratories in Australia use a cobalt-chromium welding rod, flux and an oxy-acetylene flame.⁷⁵

Applegate (1965)⁷⁶ and Asgar and Peyton (1965)¹⁶ presented a technique for casting a low fusion cobalt-chromium alloy to embedded gold wire clasps, as dental gold wires exhibit better physical properties than cast cobalt-chromium and so are able to withstand higher stresses before they are permanently deformed.

Scott and Deary (1969)⁷⁷ investigated the gold soldering of dental wrought wires to cobalt-chromium castings but the temperatures necessary caused the loss of physical properties in the wrought wires.

Brockhurst (1970)⁷⁸ states "it is recognised that some wires and gold alloys are superior to cast cobalt-chromium for retentive arms and clasps. The performance of current or improved designs can therefore benefit from improved materials. There are two ways of achieving this. Either the casting alloy may be improved, or materials with superior properties such as high strength wires or high tensile strength gold casting alloys, may be joined to the cast base."

The research done by Brockhurst showed that Nimonic 90 (cold drawn) can withstand temperatures of up to 870°C when it is gold soldered to cobalt-chromium bases.

Part 11.c.

Electrodeposition.

According to Viverto and Damiano⁷⁹ methods for electrodeposition of metals have had a dental application since 1840 and electroforming, to provide a durable die surface of copper or silver, has become a standard dental procedure^{80,81,82,83,84}.

The materials used in dentistry have been selected because of their ability to withstand corrosion in the oral environment. An alternative procedure is to cover metals that tend to corrode with a more durable coating. This can be done by electroplating.

Schwartz(1951)⁸⁵, Kazis and Kazis(1956)⁸⁶ and the United States Naval Dental School(1962)⁸⁷ recommend the use of gold plating to provide a homogeneous colour background behind acrylic facings. However electrodeposited golds being thin and lacking in hardness (approximately 70 Knoop)¹³ have been unable to protect the basis metal for any length of time.

Bien and Ayers(1958)⁵⁶ considered silver soldering of gold plated stainless steel to control crevice corrosion. The results proved unsatisfactory.

Korbelak and Duva(1961)⁸⁸ and Foulke(1963)⁸⁹ published reports on the co-deposition of gold with traces of cobalt, nickel, and indium, and electrodeposited 22K gold alloys with hardness factors up to 300 Knoop

were produced. Thus metals lacking corrosion resistance but with other desirable physical properties can be used for dental purposes protected by electrodeposited gold with a hardness factor equivalent to that of stainless steel or that of the cobalt-chromium alloys.

A procedure for gold plating cobalt-chromium dental prostheses was patented in France(1968) utilising four separate electrolytes. (ethylene glycol, sulphuric acid, hydrochloric acid and cyanide).⁹⁰

Rogers(1969)⁹¹ published an alternative technique using Woods' nickel electrolyte for both surface activation and protection prior to the electrodeposition of gold.

Rogers(1970)⁹² also advocated protecting stress-free nickel electroforms with hard, bright electrodeposited gold.

Bapna and Lugassy(1971)⁹³ found that gold plated stainless steel pins used for reinforcing amalgam restorations adhered to the amalgam matrix although there was no significant increase in the tensile strength between treated and untreated pins.

Undoubtedly, the use of techniques involving electrodeposition will expand and become increasingly important in the future as new applications are found.

Part 111.

Crevice corrosion of silver soldered stainless steel.

Stainless steel and chromium containing alloys such as Nichrome and cobalt-chromium have a high resistance to corrosion due to the passivating effect of the thin oxide layer that forms on the surface of the metal. However, this oxide layer only exists in certain environments. To obtain a tight soldered joint the fluxes used in soldering must dissolve the oxide layer to permit intimate contact between the solder and the basis metal. Removal of the protective, passive oxide layer by mechanical, chemical or electrochemical means activates the chromium-containing alloy.

The soldered joint consists of silver solder, activated stainless steel, the flux with inclusions, and passivated stainless steel beyond the joint. Crevice corrosion is initiated when an electrolyte comes in contact with the soldered joint. The breakdown of the bond between the solder and the basis metal is an electrochemical process^{94,95,96} with no evidence of macroscopic corrosion. It has been suggested that the electrochemical process is due to a cell formed by either a difference in potential between the active and passive surfaces of the joint or an oxygen concentration cell of a differential aeration type⁵⁶.

Brusse and Carmen(1934)³⁵ reported breakdown of stainless steel soldered joints when exposed to a salt-spray test and suggested the use

of a 500 fine solder, with less than one percent copper content, to reduce the effect of galvanic action.

Wilkinson (1962)⁵⁹ states "while stainless steel hooks rarely break they frequently pull away from the solder. Gold alloy hooks, on the other hand, do sometimes break where they leave the solder, but very rarely does the soldered joint itself fail. These findings were corroborated by the other two orthodontists who assisted by providing soldered joints."

Gardiner and Aamodt (1969)⁶¹ found that specimens placed in water for five weeks showed little sign of crevice corrosion. Where the edge of the solder was ground off and polished no corrosion occurred but indications of corrosion were present on the unpolished specimens.

Brockhurst (1971)⁹⁷ has found a high incidence of crevice corrosion under silver solder joints in orthodontic appliances that have been investigated after use.

Bien and Ayers (1959)⁵⁵ were able to show the presence of residual flux at the interface between the silver solder and stainless steel and consider the flux and the resultant metallic oxides to be an initiating factor in producing crevice corrosion.

To inhibit the incidence of crevice corrosion Chatfield and Swift (1949)⁴⁷ suggested the addition of 3% nickel to the silver solder. These alloys have a higher melting point and a wide melting range which

directly relates to the precipitation of carbides and also excessive softening of the wire during the soldering procedure. Bien and Ayers⁵⁶ used a solder containing 2% nickel in their investigations and Gardiner and Aamodt(1969)⁶¹ used a 3% nickel content. Crevice corrosion still occurred.

The external environment is the initiating factor in crevice corrosion. The stainless steel has an activated surface under the silver solder and a passivated surface beyond the solder joint, but the passivated surface is only relative and can alter. The activated stainless steel would be anodic to the silver solder forming a corrosion cell and so could be expected to corrode. This corrosion at the interface between the stainless steel and the silver solder would form a crevice and therefore an oxygen concentration cell. If the solder joint was protected to prevent ionisation then crevice corrosion should not occur.

Bien and Ayers(1958)⁵⁶ investigated the use of gold plated stainless steel as they thought that the continuous passive film of gold should have a greater chemical stability than the oxide film of stainless steel. However, their results after subjecting specimens to accelerated corrosion tests did not confirm this concept.

Due to the feasibility of this principle a broader investigation was planned utilising a technique developed by Rogers(1969)⁹¹ for the electro-deposition of gold on cobalt-chromium alloys.

Objects of the investigation.

It was felt that an investigation should be commenced to determine the types of bonding or joining that occurred between metals so that consideration could be given, if possible, to improving the bond or preventing its failure by an understanding of some of the factors associated with the bonding processes.

Scope of the investigation.

Although many investigations have been carried out on the physical properties of metal unions used in dentistry, little metallographic evidence has been presented regarding the type and nature of these unions.

In this investigation metallographic techniques were developed to examine the interface existing between some of the metals used in dentistry and the following interfaces were examined.

Type B gold alloy	-	24K electroformed gold
Type C gold alloy	-	24K electroformed gold
Gold solder	-	gold alloy
Silver solder	-	stainless steel
Gold solder	-	stainless steel
Electroplated gold	-	stainless steel
Gold solder	-	cobalt-chromium alloy
Electrodeposited gold	-	cobalt-chromium alloy
Resistance welded stainless steel.		

OBJECTS AND SCOPE OF THE INVESTIGATION.

A requirement noted for metals and alloys used for dental purposes is the resistance to tarnish and corrosion in the oral environment.

The two basic groups of alloys are (i) the precious metal alloys and (ii) the chromium containing alloys. The surface behaviour of the alloys when soldered or electroplated is related to their resistance to chemical attack, so for the purpose of this investigation of metal to metal interfaces, the following classification will be used.

Precious metal alloys.

- i.e. a. electroformed gold - gold alloy interface
- b. gold solder - gold alloy interface

Chromium containing alloys.

- i.e. a. gold solder - cobalt-chromium alloy interface
- b. electroplated gold - cobalt-chromium alloy interface
- c. silver solder - stainless steel interface
- d. gold solder - stainless steel interface
- e. electroplated gold - stainless steel interface
- f. resistance welded stainless steel interface.

The investigation was carried out in three parts.

Part 1. Precious metal alloys.

- a. Metallographic examinations and hardness testing were carried out to determine whether bonding had occurred between the cast gold and the electroformed gold matrix. The influence of

casting conditions on the bond was also investigated.

b. A metallographic examination on the bond between gold and gold solder was also carried out.

Part 11. Chromium containing alloys.

A metallographic examination was carried out on the type of bond occurring between these alloys, gold solder and electroplated gold. The interface between silver soldered stainless steel and resistance welded stainless steel were also investigated.

Part 111. Crevice corrosion of silver soldered stainless steel.

Accelerated corrosion tests were carried out to determine methods of controlling crevice corrosion of silver soldered stainless steel joints.

MATERIALS AND METHODS.

PART 1. PRECIOUS METAL ALLOYS.

MATERIALS AND METHODS.

Part 1.a.

The electroformed gold - gold alloy interface.

Electroformed gold matrices were produced by the method described by Rogers and Armstrong.¹³ Briefly this method is as follows. Gold matrices were electroformed on metallised hydrocal dies as described by Rogers,¹⁴ simulating M.O.D. restorations.

The electrolyte used had the following formula.

Potassium gold cyanide	14.1g
Potassium cyanide	18.3g
Potassium carbonate	14.1g
Boric acid	11.4g
Distilled water	to 1 litre

At an operating temperature of 65°C and a current density of 3.2 amp per square decimeter this electrolyte has a rate of deposition of 0.1 to 0.12 mm./hour. Agitation is necessary and a gold anode is used. Time of electrodeposition is two hours.

Quantities of four types of inlay gold were purchased. The details relating to composition and properties were supplied by the manufacturers.

B⁽ⁱ⁾ Precious Metal Refining Co. Type B.

Au 83.36 per cent

Ag 8.34 per cent

Cu 8.3 per cent

Melting Point - 935°C (Liquidus)

Hardness (Vickers) 94 quenched
100 hardened.

B⁽ⁱⁱ⁾ S.S. White. Type 820.

Au 79.0 per cent

Ag 8.4 per cent

Pd 3.0 per cent

Ir 0.1 per cent

Cu 8.0 per cent

Zn 1.5 per cent

Melting Range $890\text{--}1014^{\circ}\text{C}$

Hardness (Brinell) 96 quenched.

C Precious Metal Refining Co. No.3.

Au 78.8 per cent

Ag 7.0 per cent

Pt 3.0 per cent

Pd 2.3 per cent

Cu 7.8 per cent

Zn 1.1 per cent

Melting Point - 960°C (liquidus)

Hardness (Vickers) 120 quenched
142 hardened.

D S.S. White. No.3.

Au 70.08 per cent

Ag 11.0 per cent

Pt 4.90 per cent

Ir 0.02 per cent

Cu 13.50 per cent

Zn 0.5 per cent

Melting Range $862\text{--}960^{\circ}\text{C}$

Hardness (Brinell) 153 quenched
238 hardened.

After preliminary experimentation,¹³ 30 M.O.D. specimens were prepared by waxing up the electroformed gold matrices using an inlay wax conforming to the Australian Standard T14⁰ by the technique described by Rogers.¹⁴

Spruing.

Ten waxed up patterns were sprued both on the mesial and distal marginal ridges with 14 gauge (1.7mm.) hollow stainless steel sprue formers.

The remaining twenty patterns were sprued with single 10 gauge (2.6mm.) hollow stainless steel sprue formers with 6mm. diameter reservoirs placed 3mm. above the pattern.

Investing.

The investing crucibles were lined with wet asbestos and the patterns were centrally positioned so that their highest point was 6mm. below the rim of the crucible. A cristobalite investment was used conforming to the Australian Standard T22.⁺

The double-sprued specimens were invested using an electrical vibrator and the remaining were vacuum invested using a Whipmix Power Mixer with Vac-u-mixer. A surface tension reducing agent was used on all patterns.*

0 Kerr. Blue Inlay Casting Wax. Regular.

+ Kerr. Cristobalite Investment.

* Kerr Vacu-Film for vacuum investing and Kerr Debubblizer on the remainder.

Casting.

The ten double-sprued specimens that were invested with the vibrator were heated to a mould temperature of 700°C in an electric furnace(Fig.1).

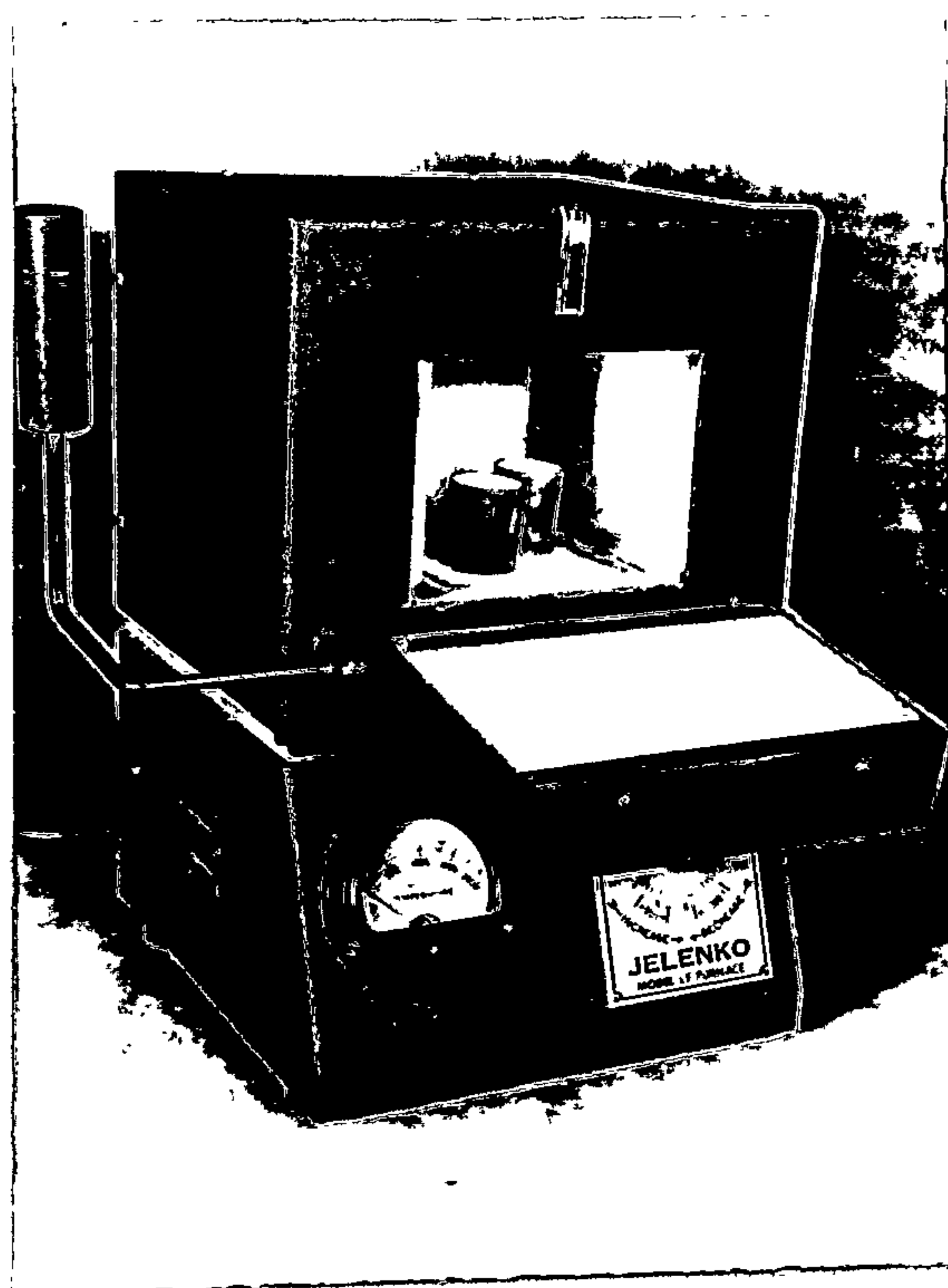


Figure 1. Electric Furnace.

The reducing zone of a gas air flame was used to melt the alloys and the casting was carried out in a hand-swung bucket.

Five castings were done in type B⁽ⁱ⁾ gold and five in type C gold.

Of each of these groups of five specimens flux was added to three of the matrices prior to waxing up to evaluate its effect during the casting procedure.

The flux used was borax glass that had been ground and made into a paste with distilled water. One of the non-fluxed specimens was stored in the furnace for 24 hours at 700°C prior to quenching.

The remaining specimens were cast using the Jelenko Thermotrol(Fig.2)

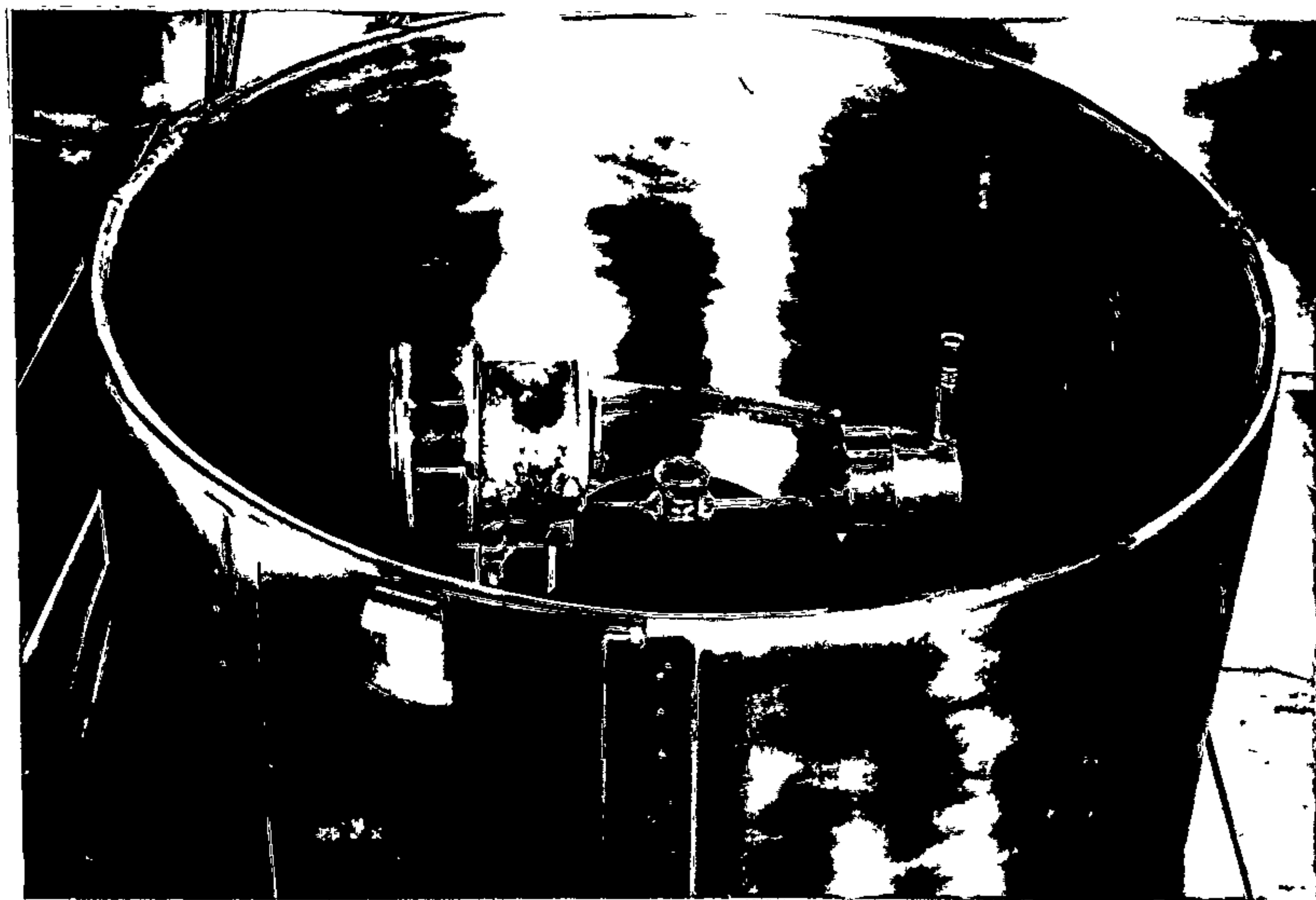


Figure 2. Jelenko Thermotrol casting machine.

Figure 2. Jelenko Thermotrol casting machine.

which consists of a small thermostatically controlled electric furnace utilising a carbon crucible mounted on a rotating arm. The casting is accomplished by centrifugal means.

Six of the remaining sixteen specimens were heated to mould temperatures of 550°C , 650°C and 750°C and cast in a Jelenko Thermotrol casting machine in type B⁽ⁱ⁾ gold and cast at 20°C and 50°C above the melting point of the gold alloy. A further six specimens were similarly cast in type C gold.

Eight specimens were heated to a mould temperature of 750°C and cast in a Jelenko Thermotrol - four in type B⁽ⁱⁱ⁾ gold at a casting temperature of 1015°C and four in type D gold at a casting temperature of 960°C .

After bench cooling for five minutes, the cast inlays were quenched in water, removed from the investment and pickled by boiling in 50% hydrochloric acid.

The specimens were sectioned and mounted in a cold curing resin used for metallographic purposes (Fig. 3A).

A conductive attachment for electrolytic etching was provided by drilling a hole through the back of the resin to contact the specimen. This hole was tapped to accept an $\frac{1}{8}$ inch brass metal thread. To ensure an effective electrical connection between the thread and the specimen a small quantity of gold foil was placed into the prepared hole and then

the brass thread was screwed firmly into position, forcing the gold foil against the gold specimen(Fig.3B).

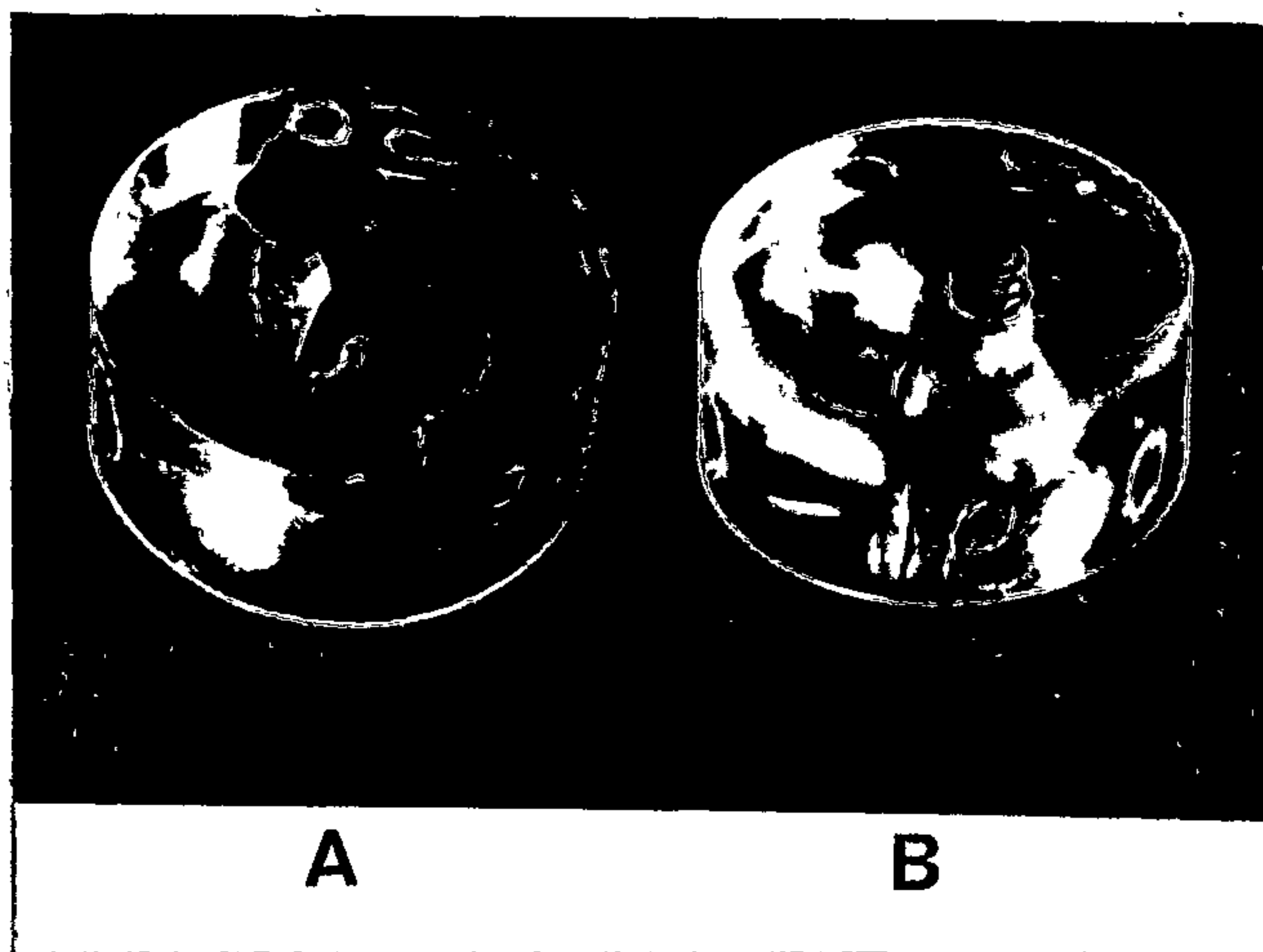


Figure 3. Specimens mounted in cold curing resin for metallographic examination.

The initial surface preparation was carried out on a Lunn Pregrinder(Fig.4) using 220,320,400 and 600 mesh abrasive papers.

The specimens were cleaned ultrasonically for five minutes in a non-ammoniated general purpose cleaner* to remove any abrasive particles and then polished for three minutes using a 7 μ diamond paste with an emulsified oil lubricant on a D.P. Mol-cloth pad rotating at 250 r.p.m.

The specimens were cleaned again and repolished using a medium

* L. & R. Denson Dental Ultrasonic Solution. General Purpose Cleaner.

Non-ammoniated.

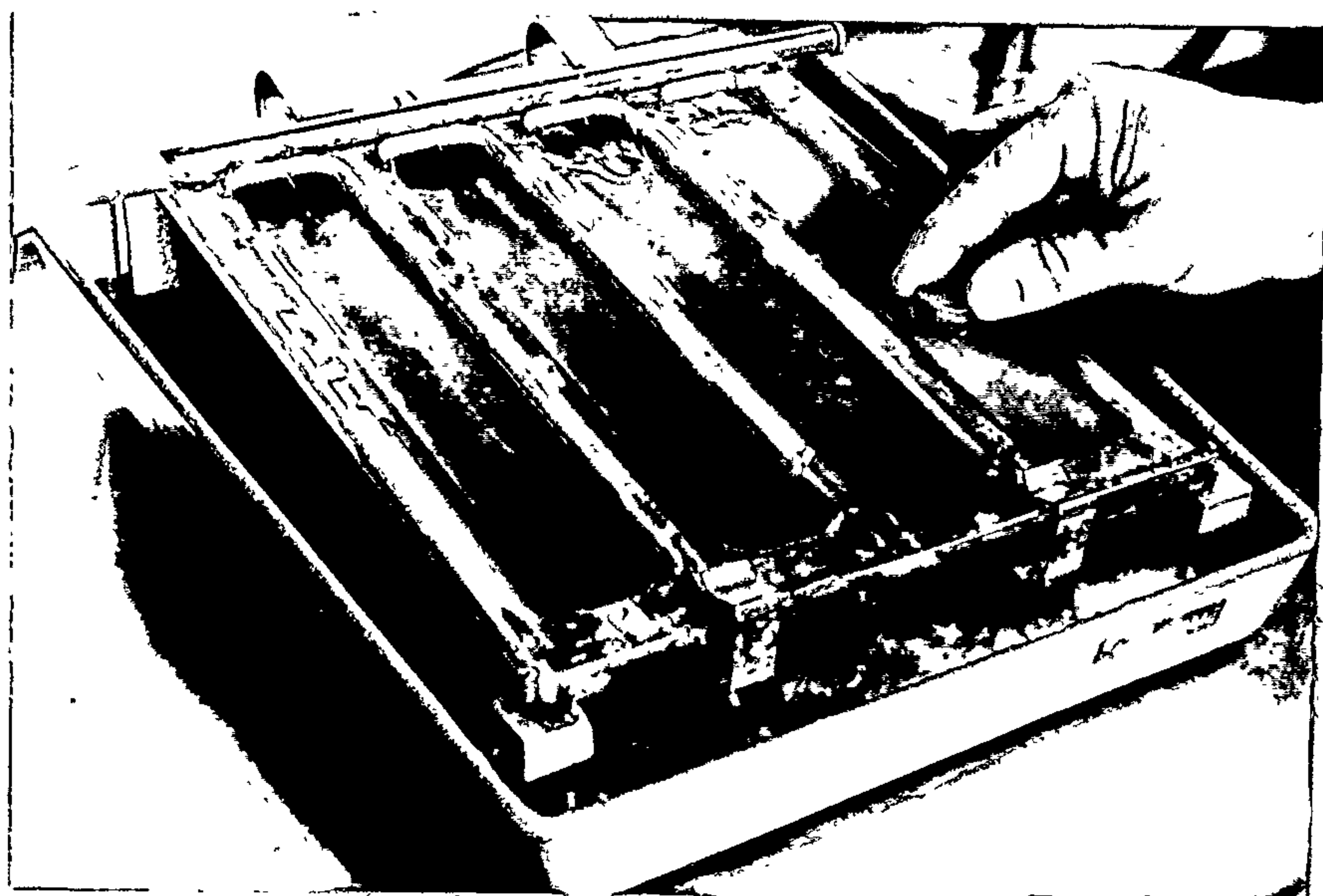


Figure 4. Lunn Pregrinder.

grade final polish diamond paste. After washing and drying the specimens were polished electrolytically on a Struers Relopol (Fig. 5).

The Struers Relopol is an electrolytic lap-polishing machine used for electropolishing metallographic preparations of gold, platinum and silver and similar soft metals which are extremely sensitive to mechanical treatment. It has the advantage of producing distortion-free surfaces without the disadvantage of forming the relief structures which can be caused by anodically dissolving less noble phases in heterogeneous structures.

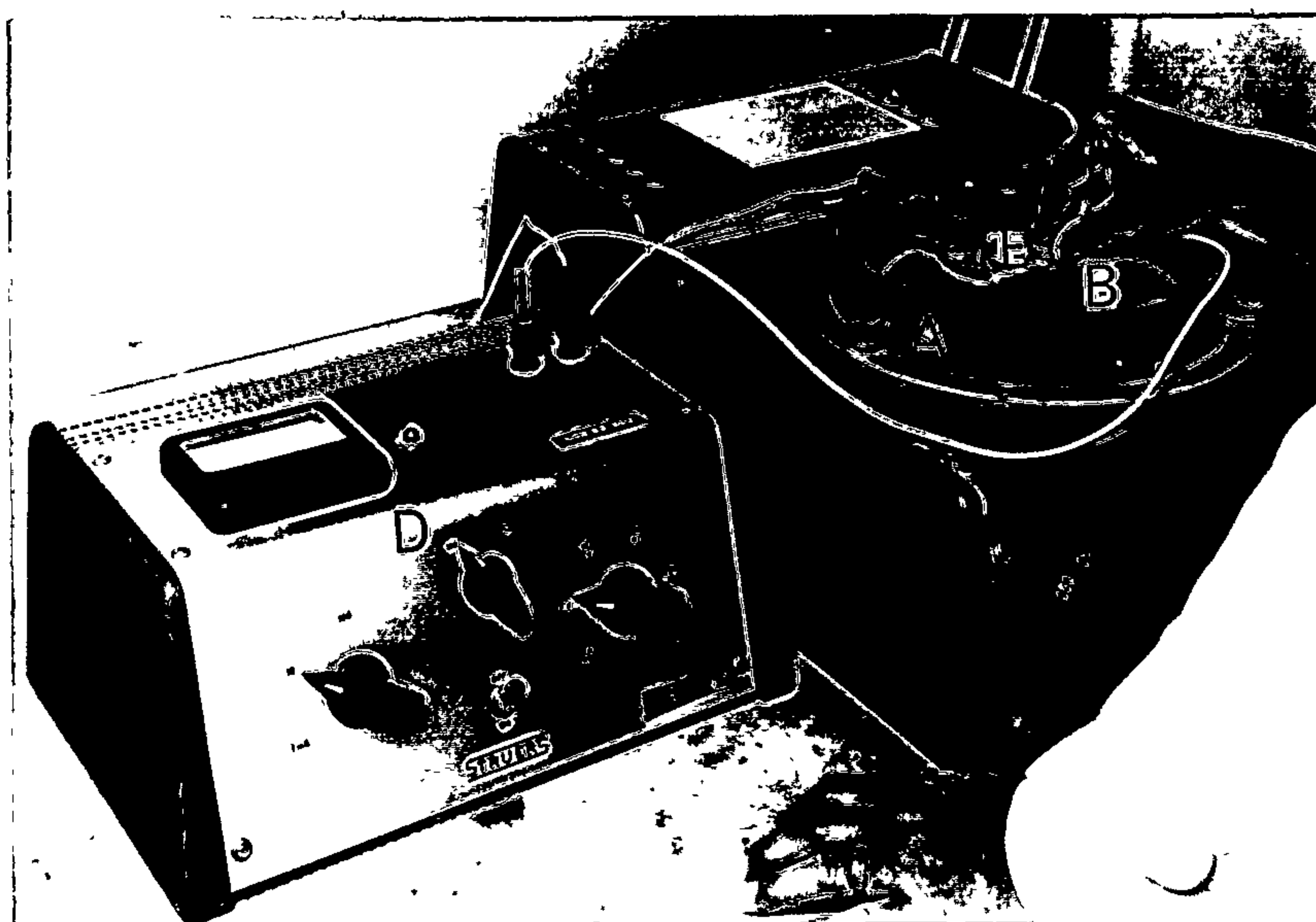


Figure 5. Struers Relopol used for electrolytic polishing of specimens.

The method uses simultaneous mechanical polishing to support the anodic effect. Figure 6 shows the principal components of this equipment.

- A. The ring which supports the electrolyte baffle and carries the cathode contact.
- B. Rotating stainless steel polishing disc fitted with a Reinacher-cloth which is woven from soft acid resisting fibres.
- C. Electrolyte baffle plate which returns the centrifugated electrolyte back to the centre of the polishing disc.
- D. Power control unit for the supply of either alternating or direct current.

E. Anodic specimen fitted with electrical contact.

The RP3 electrolyte had the following composition.

Potassium rhodanid	20g
Ammonium chloride	20g
Ammonium triosulphate	20g
Water	1 litre

A small quantity of Microid Alumina^{*} was added to the polishing pad and a current density of 1.5amp/dm.² was applied for periods of up to three minutes.

The specimens were rewashed and dried and subjected to etching procedures.

Initially the following chemical etchants were used.

- a. 50% aqua regia
- b. 50% tinc. iodine in potassium iodide
- c. 5% potassium bromate, 5% potassium bromide and hydrochloric acid.

Electrolytic etching using the external etching apparatus of the Disa

Electropol was then carried out(Fig. 6).

* Griffin and George.

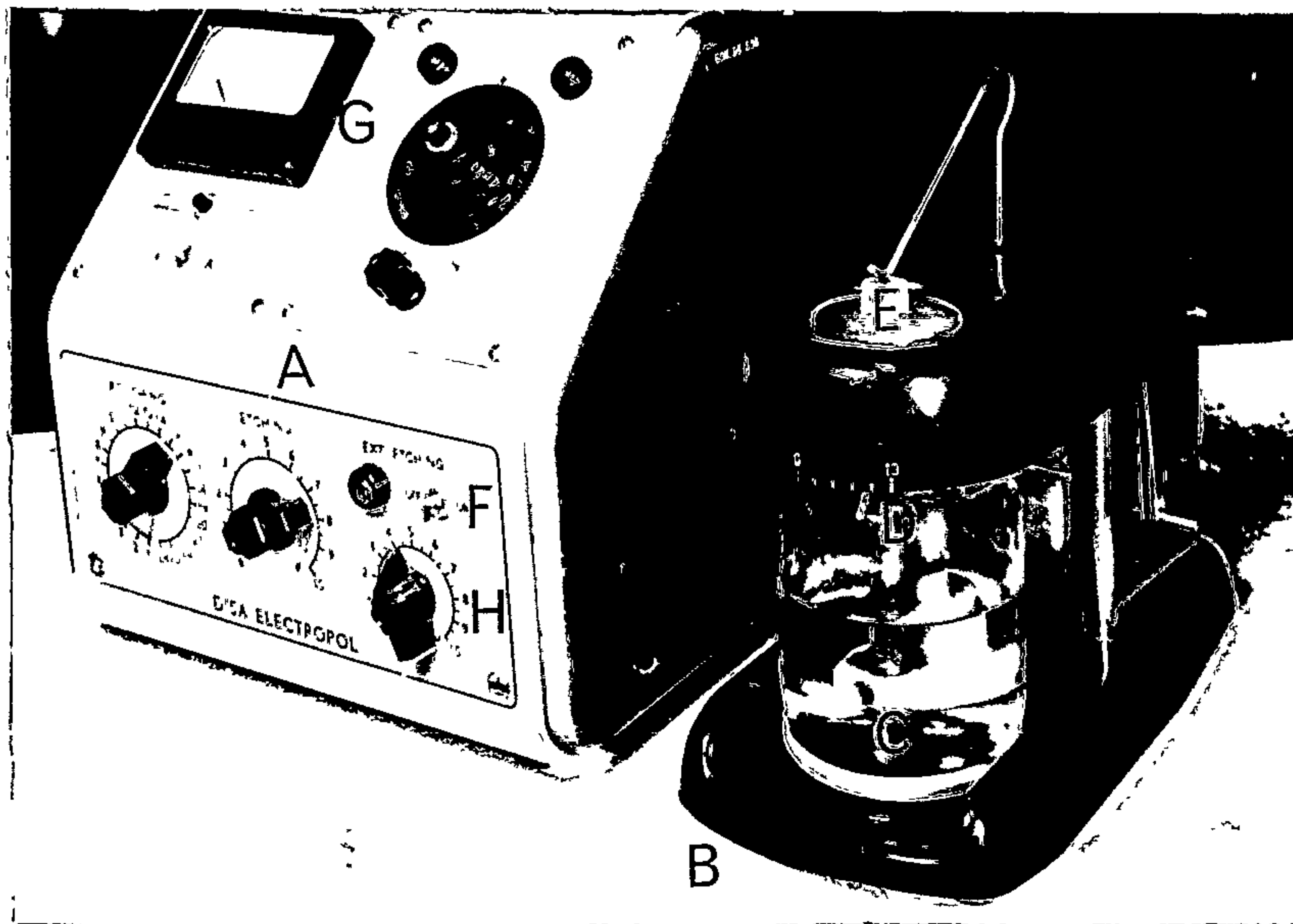


Figure 6. Disa Electropol used for electrolytic polishing and etching procedures.

The Disa Electropol consists of a power unit (A) supplying both polishing and etching current to the flow through the electrolytic equipment (B). The electrolyte is placed in the reservoir bowl (C) and is pumped at a controlled flow rate, determined by the flow rate regulator (D), between the anodic specimen (E) and a stainless steel cathode.

Various proprietary electrolytes (composition undisclosed) are produced by Struers for various metals and alloys.

The Disa Electropol has a built in unit for external etching. This

apparatus is not shown in figure 7, but it consists of a stainless steel basin and tweezers connected to a plug which is placed into the operating power supply. When the external etching apparatus is switched on (F), the flow through the system and the timer are cut out of circuit. However the meter (G) is still connected for reading of voltage and amperage as required. The stainless steel basin is cathodic and the specimen with its electrical connection held by the tweezers becomes anodic when placed in the chosen electrolyte. The current is controlled by the regulator (H).

The following electrolytes were used:

- | | | |
|----|---------------------|------------|
| a. | Silver cyanide | 35g. |
| | Potassium cyanide | 70g |
| | Potassium carbonate | 40g |
| | Distilled water | to 1 litre |
| b. | Potassium cyanide | 10% |

A current density of 1.2 amp/dm.^2 was applied from 8 to 12 seconds.

Microscopical examination and photomicrography were carried out on a Leitz Panphot microscope(Fig.7).

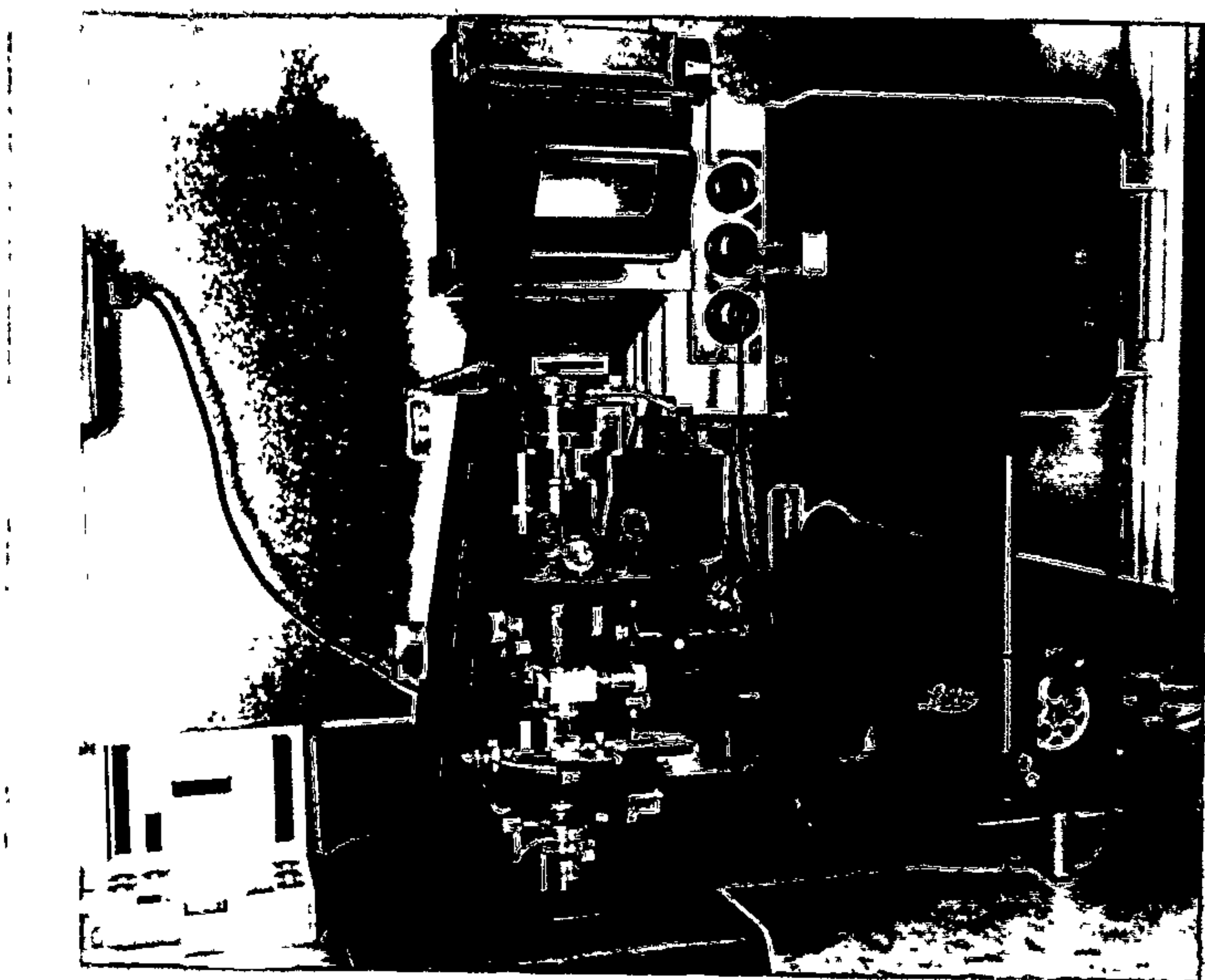


Figure 7. Leitz Panphot microscope.

Microhardness testing was carried out on ten of the mounted specimens using a Leitz "Durimet" microhardness testing machine, (fig.8).

Each specimen was placed on the table of the "Durimet" and positioned so that the diamond indenter would be placed into the electroformed gold matrix, the interface between the matrix and the cast gold, and three underlying areas within the cast gold as shown in figure 41.

The diamond takes fifteen seconds to reach the specimen and it was left in contact with the specimen for an additional ten seconds. The resulting pyramidal-shaped indentation was measured across

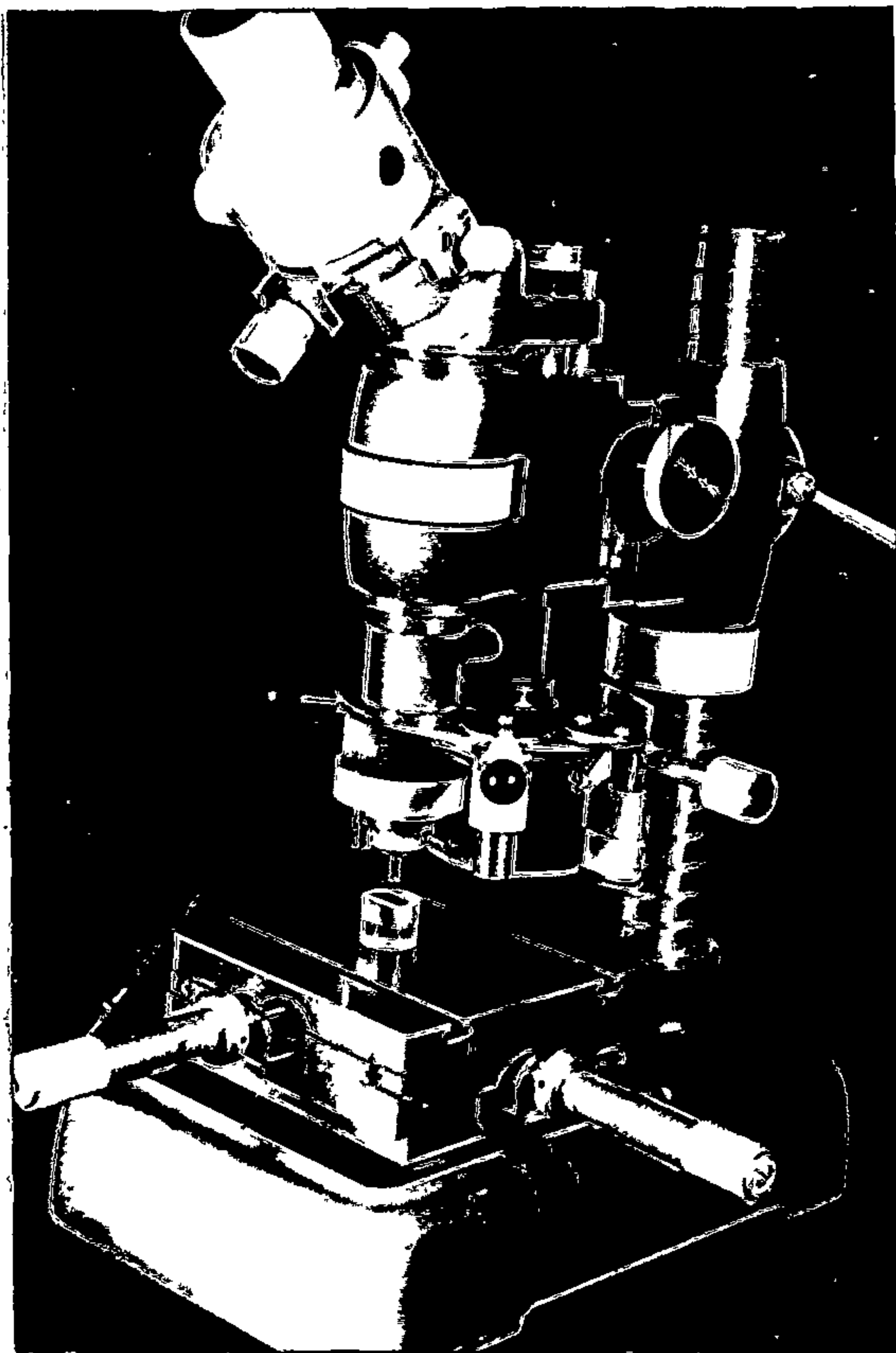


Figure 8. Leitz Durimet microhardness testing machine.

both of its diagonals utilising the microscopic attachment coupled to the high power lens.

The hardness was determined by averaging the diagonal measurements and relating this to the table appropriate to the load used on the diamond. In this instance a 100 gram load was used.

Part 1.b.

The gold solder - gold alloy interface.

Quantities of wrought platinised gold wire and 18K gold solder were purchased and they had the following composition and properties.

17 gauge platinised gold wire*

Gold	75 per cent
Silver	10 per cent
Copper	15 per cent
Melting range	940-960°C

To this total 10 per cent platinum was added.

18K gold solder(easy)*

Gold	75 per cent
Silver	8 per cent
Copper	3 per cent
Zinc	7 per cent
Cadmium	7 per cent
Melting range	739-752°C.

The wire was cut into 2cm. lengths and a jig(Fig.9) was constructed to hold the wire at right angles to form a symmetrical "T" joint.

Twenty soldered joints were constructed using a Gruenburg burner and borax glass flux. Ten specimens were quenched immediately and ten were held in the flame for five seconds and then allowed to bench cool.

* Precious Metal Refining Co.

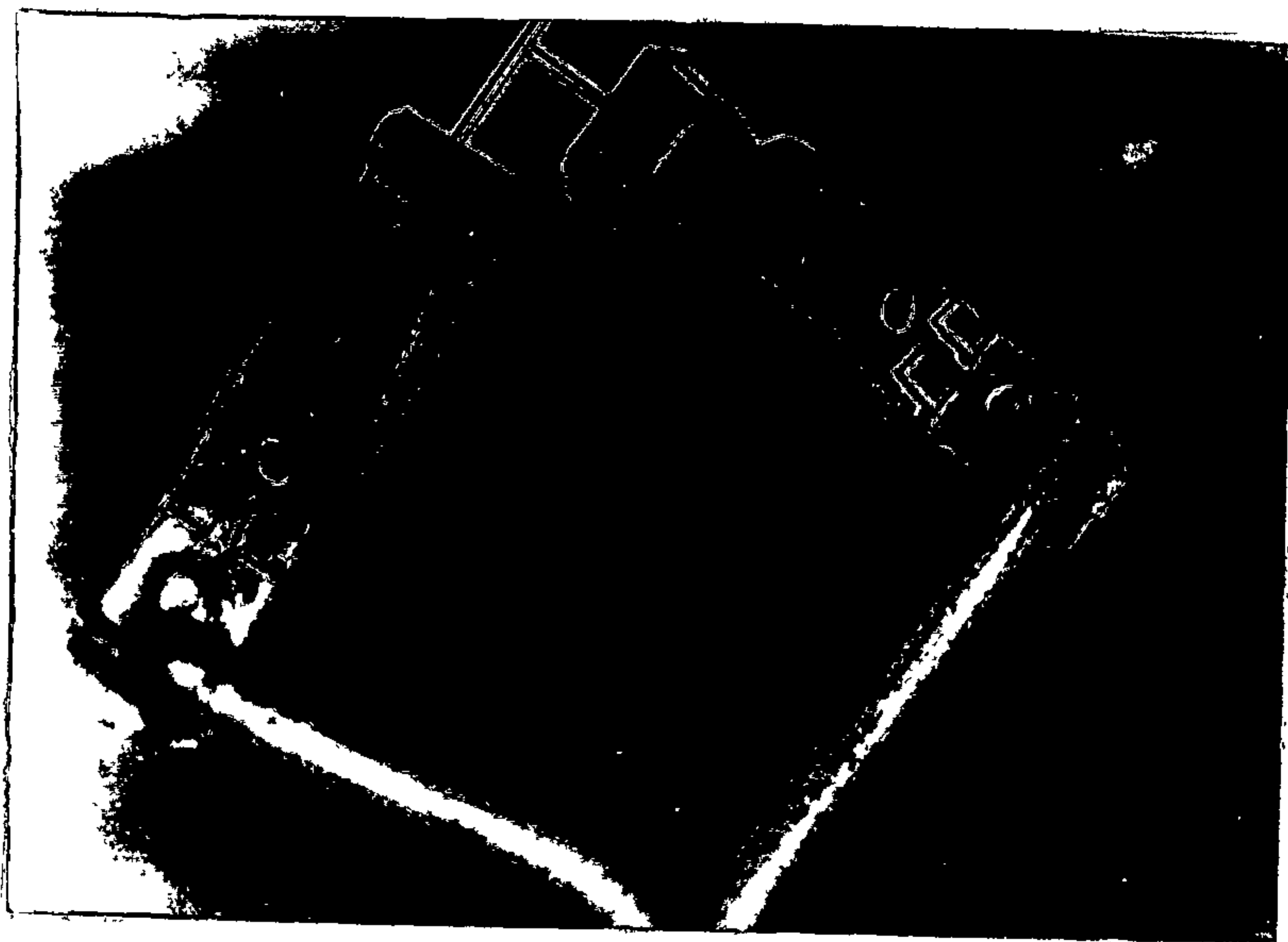


Figure 9. Jig used for holding the wire specimens for soldering procedures.

One and a half centimeters of the long arm of the "T" joint was bent so as to protrude from the back of the mounting resin, thus forming an electrical contact. Two sections, five millimeters long, on each side of the top bar of the "T" were bent at right angles to produce a standard size joint and to assist retention of the joint in the resin(Figure 10).

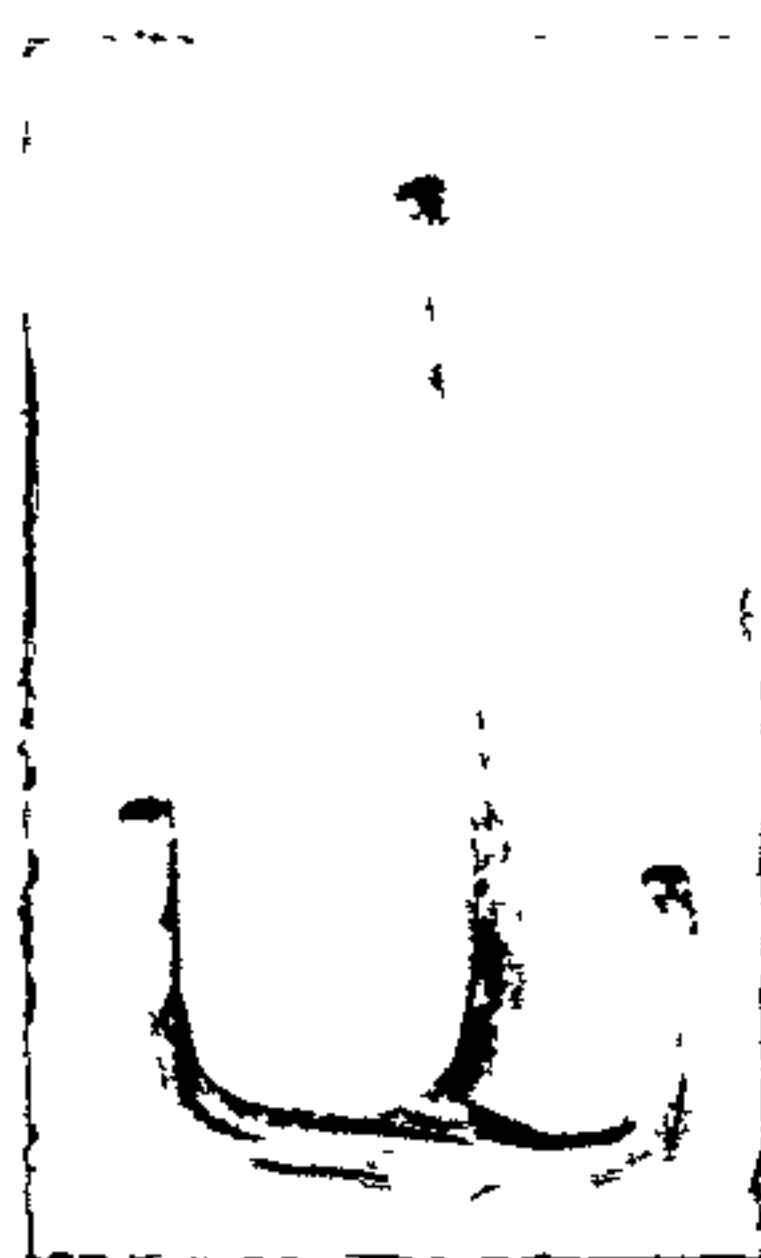


Figure 10. Soldered wire specimen prior to mounting in cold
curing resin.

Mounting , polishing and etching of the specimens for metallographic examination was the same as previously described in Part 1.a.

RESULTS.

PART 1. PRECIOUS METAL ALLOYS.

RESULTS.

Part 1.a.

The electroformed gold - gold alloy interface.

TABLE I.

Investing and casting conditions for specimens in figures 11 to 19.

Ten double-sprued inlays, vibrator invested and cast by using a gas-air flame and a hand-swung bucket.

Gold Alloy	Mould Temp °C	Casting Temp.	Figure
3 Type B ⁽ⁱ⁾ . Interface fluxed.	700	gas-air flame used	* 11,12
3 Type C. Interface fluxed.	"	"	13*,14
2 Type B ⁽ⁱ⁾ .	"	"	15
1 Type C.	"	"	16*,17
1 Type C replaced in furnace for 24 hours after casting.	"	"	18,19

All figures are electrolytically polished and etched except those marked *.

TABLE II.

Investing and casting conditions for specimens in figures 20 to 35.

Twenty single sprued inlays, vacuum invested and cast using a Jelenko Thermotrol casting machine.

Gold Alloy	Mould Temp. °C	Casting Temp.	Figure
Type B.	550	955	20
"	"	985	21
Type C.	"	980	22
"	"	1010	23
Type B ⁽ⁱ⁾ .	650	955	24
"	"	985	25
Type C.	"	980	26
"	"	1010	27
Type B ⁽ⁱ⁾ .	750	955	28
"	"	985	29
Type C.	"	980	30
"	"	1010	31
4 x Type B ⁽ⁱⁱ⁾ .	"	1015	* * 32-33
4 x Type D.	"	960	* * 34-35

All figures are electrolytically polished and etched except those marked *.

TABLE III

Vickers hardness of electroformed gold matrix.

Type C gold.				Type D gold.		
Load in gm.	15	50	100	15	50	100
Mean VHN (10 tests.)	46.1	53.1	41.5	49.2	42.5	40.9
Standard deviation.	4.9	6.7	2.9	5.8	3.1	3.4
95% confidence level.	49.2	57.3	43.3	52.8	44.5	43.1
	43.0	48.9	39.6	45.5	40.6	38.8
99% confidence level.	50.8	59.5	44.2	54.7	45.4	44.1
	41.5	46.8	38.7	43.6	39.6	37.7

Vickers hardness at interface

Type C gold.				Type D gold.		
Load in gm.	15	50	100	15	50	100
Mean VHN (10 tests.)	56.1	60.7	52.0	69.8	67.5	56.4
Standard deviation.	12.5	8.8	9.4	21.0	4.2	4.6
95% confidence level.	64.0	66.2	57.9	83.1	70.2	59.9
	48.2	55.2	46.0	56.5	64.9	54.0
99% confidence level.	68.0	69.0	60.9	89.7	71.6	61.3
	44.2	52.4	43.1	49.9	63.5	52.5

Vickers hardness of cast gold alloy.

Type C gold.				Type D gold.		
Load in gm.	15	50	100	15	50	100
Mean VHN (10 tests.)	136.9	153.6	144.4	179.2	158.2	163.1
Standard deviation.	27.0	3.8	7.6	25.4	18.3	3.9
95% confidence level.	154.0	156.0	140.2	195.3	169.8	165.5
	119.8	151.1	140.0	163.1	146.6	160.7
99% confidence level.	162.5	157.2	151.6	203.3	175.6	166.8
	111.2	150.0	137.2	155.1	140.8	159.4

Figure 11(x 10). Etched in aqua regia.

Conditions.

Mould temperature 700°C

Gas-air flame

Type Bⁱ gold alloy.

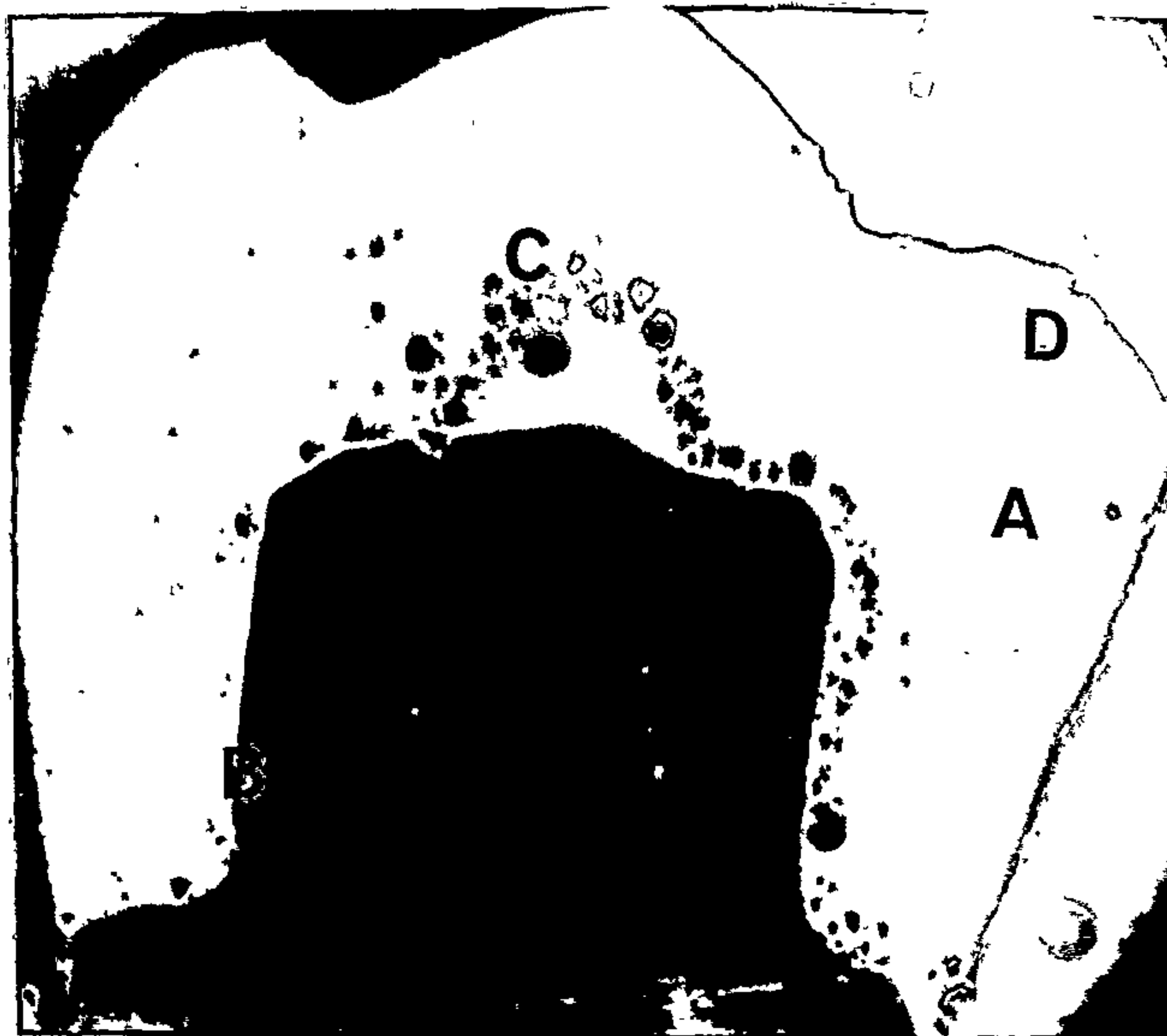
Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to flux (?)

D. Pinhole porosity.



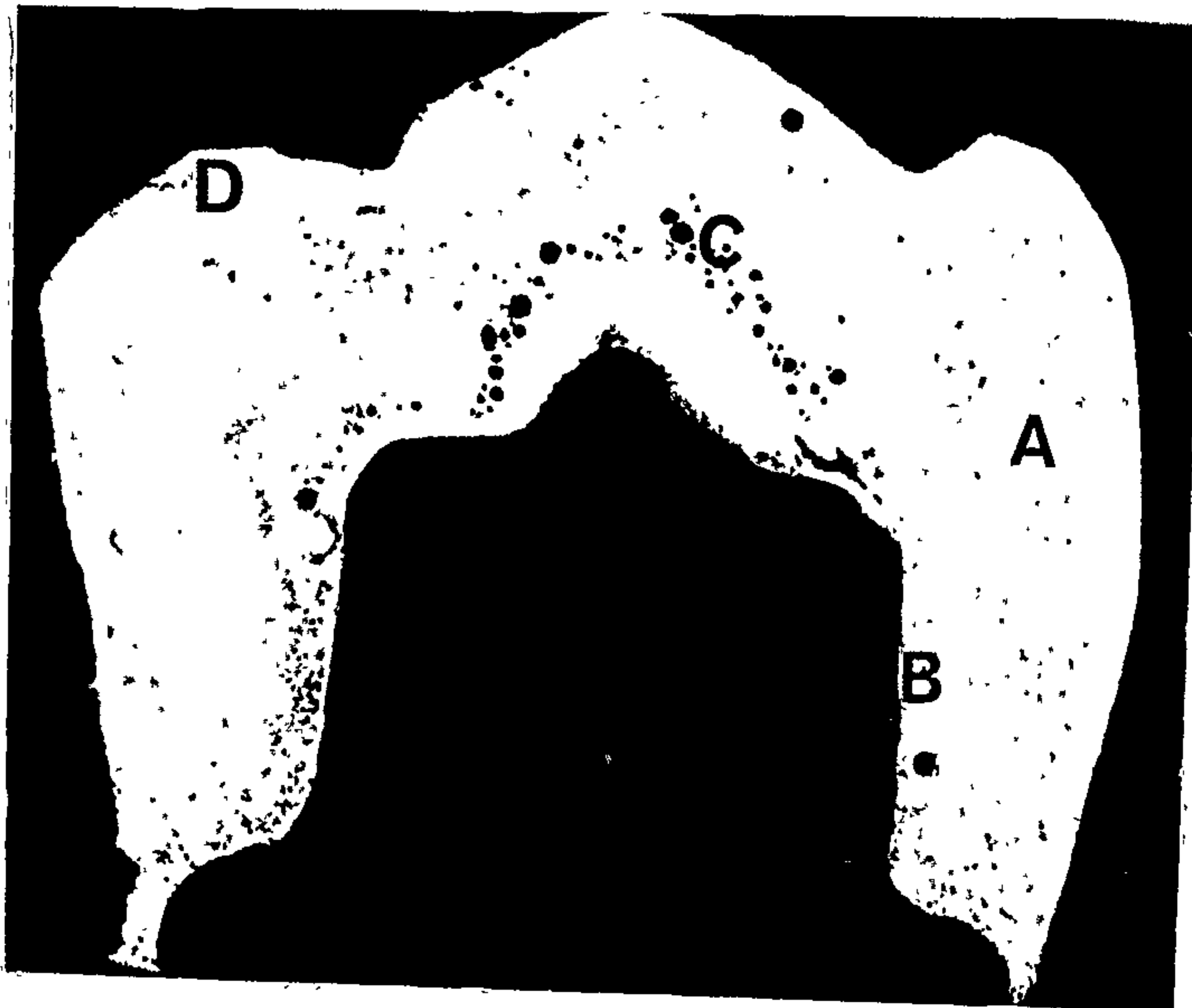


Figure 12(x 10). Electrolytically etched.

Same specimens as figure 11. - (mirror image).

Legend.

- A. Cast gold
- B. Electroformed gold matrix
- C. Porosity due to flux (?)
- D. Dendritic structure of porosity indicates microporosity.

Figure 13(x 10). Etched in aqua regia.

Conditions.

Mould temperature 700°C

Gas-air flame

Type C alloy.

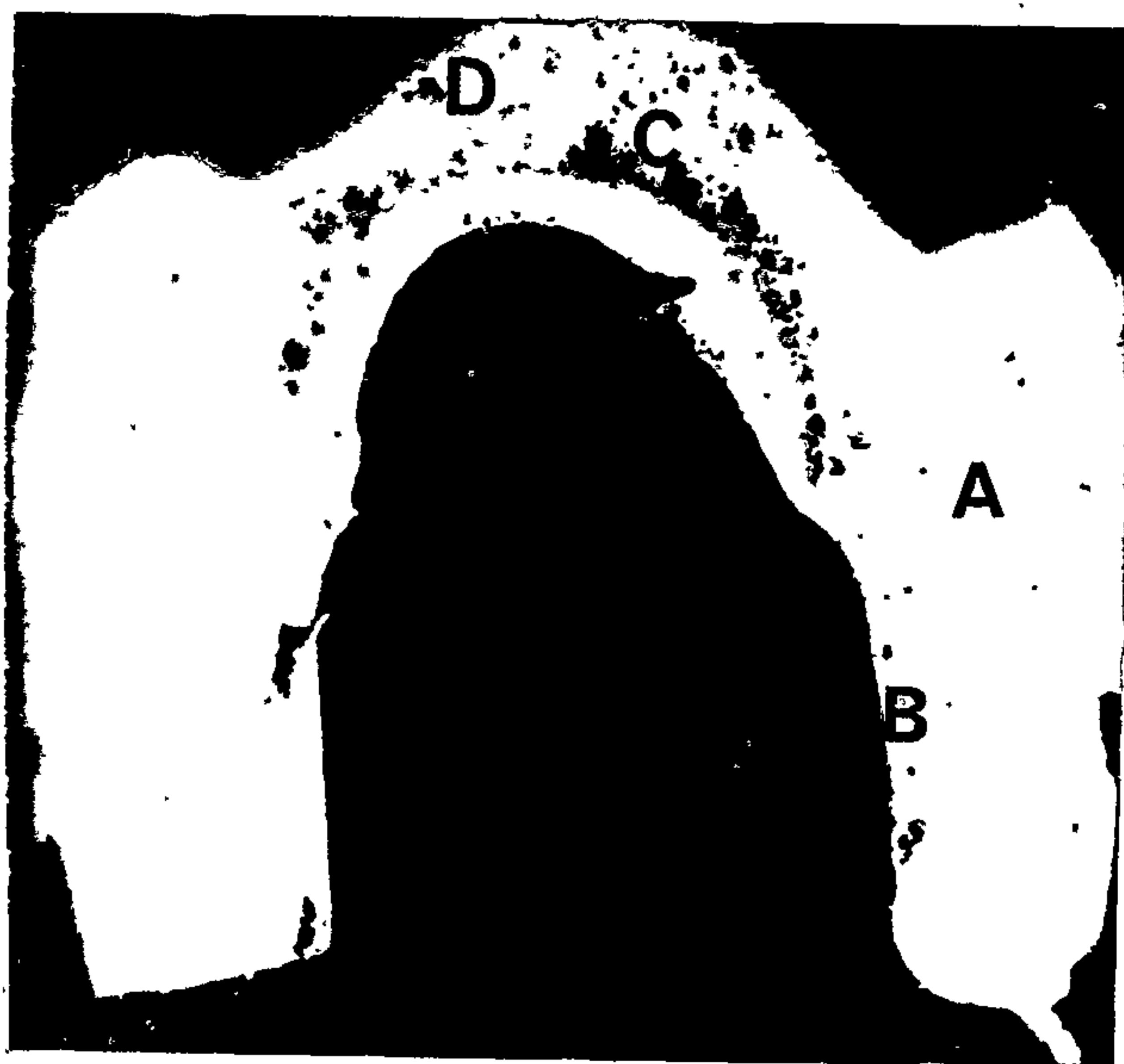
Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to flux

D. Pinhole porosity (?).



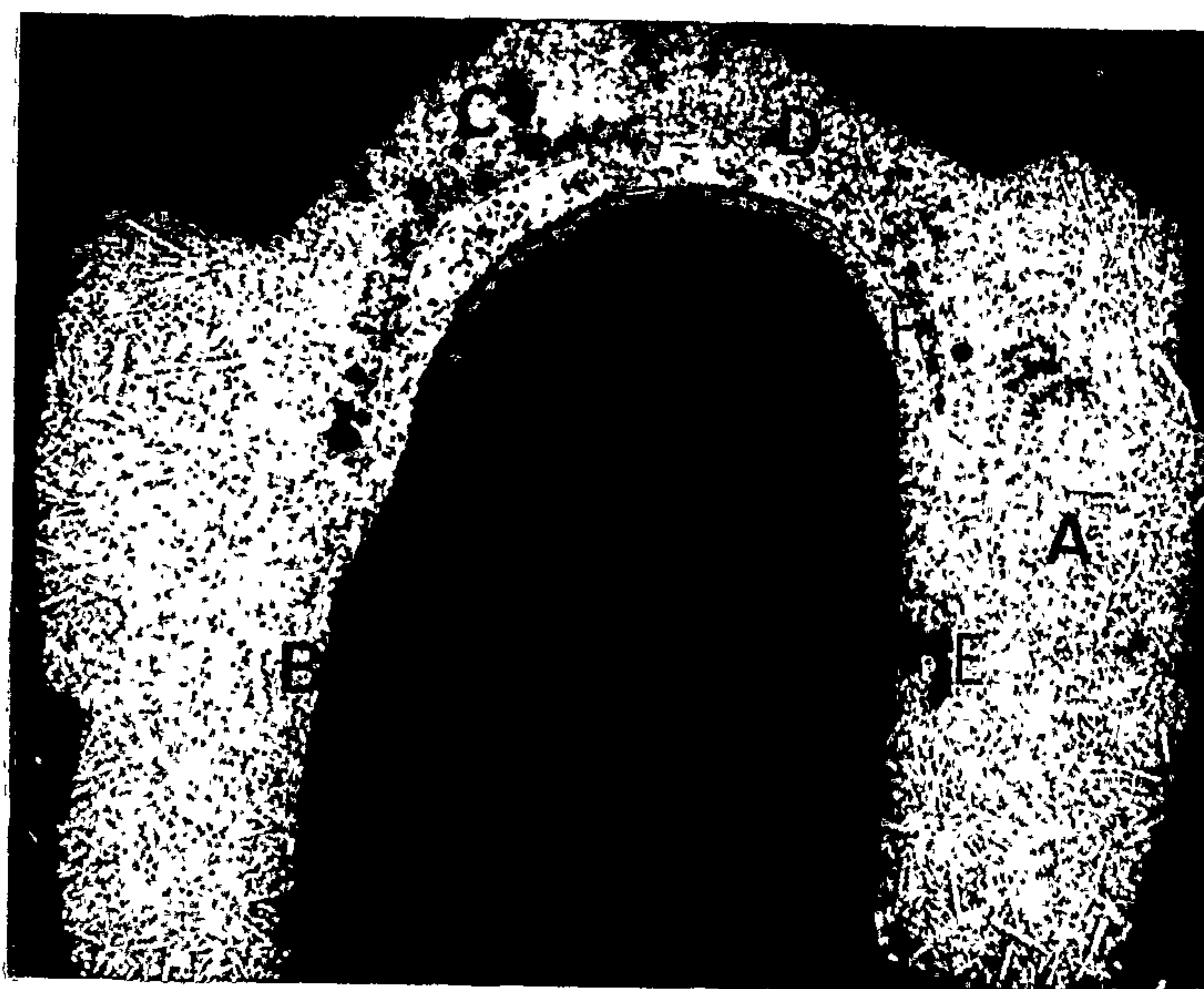


Figure 14(x 10). Electrolytically etched.

Same specimen as figure 13. - (mirror image).

Legend.

- A. Cast gold
- B. Electroformed gold matrix
- C. Porosity due to flux
- D. Dendritic structure of porosity indicates microporosity
- E. Matrix perforated
- F. Gold under matrix.

Figure 15(x 10). No flux added to matrix.

Conditions.

Mould temperature 700°C

Gas- air flame

Type Bⁱ alloy.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to gas inclusion.

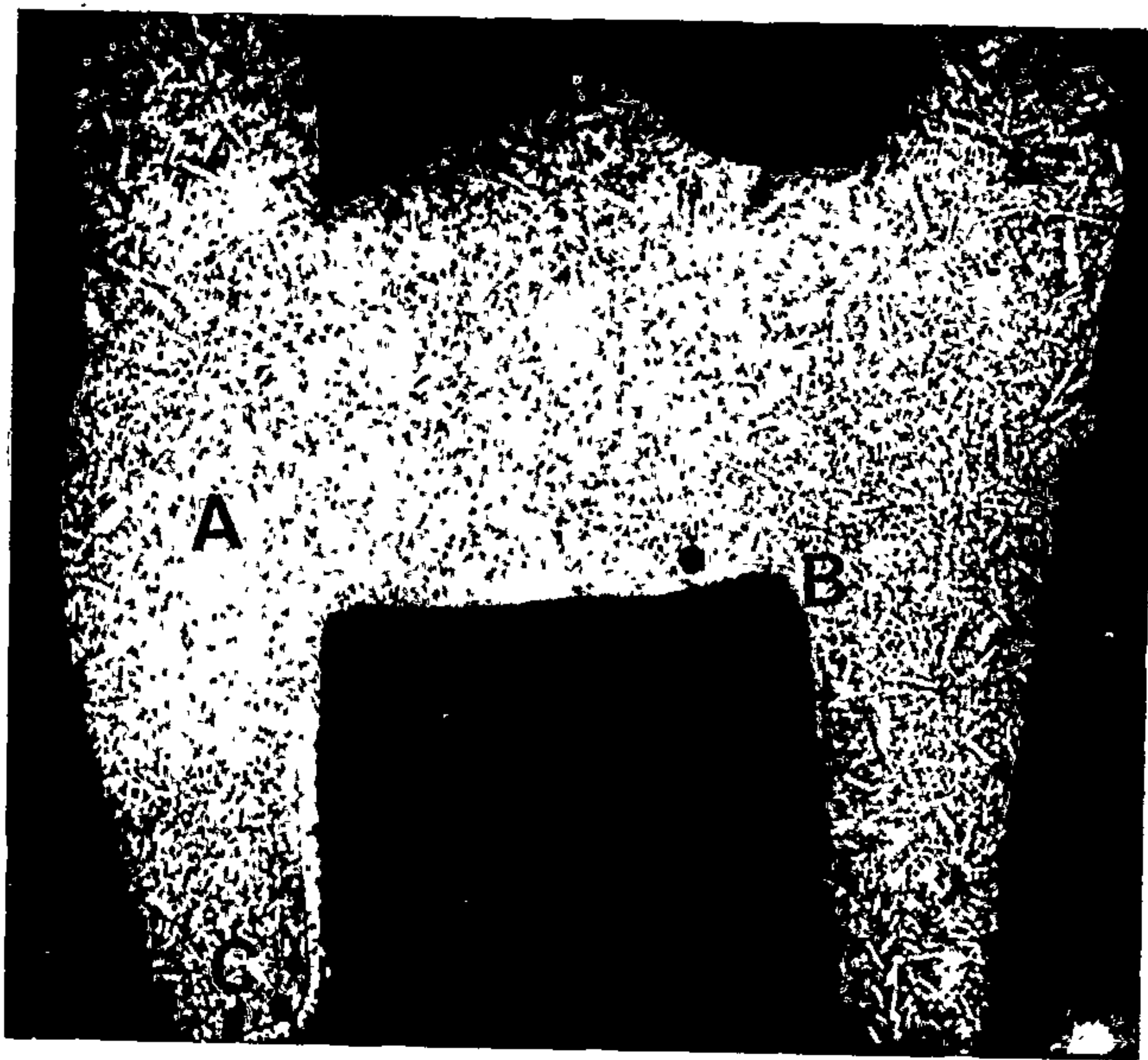




Figure 16(x 8). Etched in aqua regia.

Conditions.

Mould temperature 700°C

Gas - air flame

Type C alloy.

Legend.

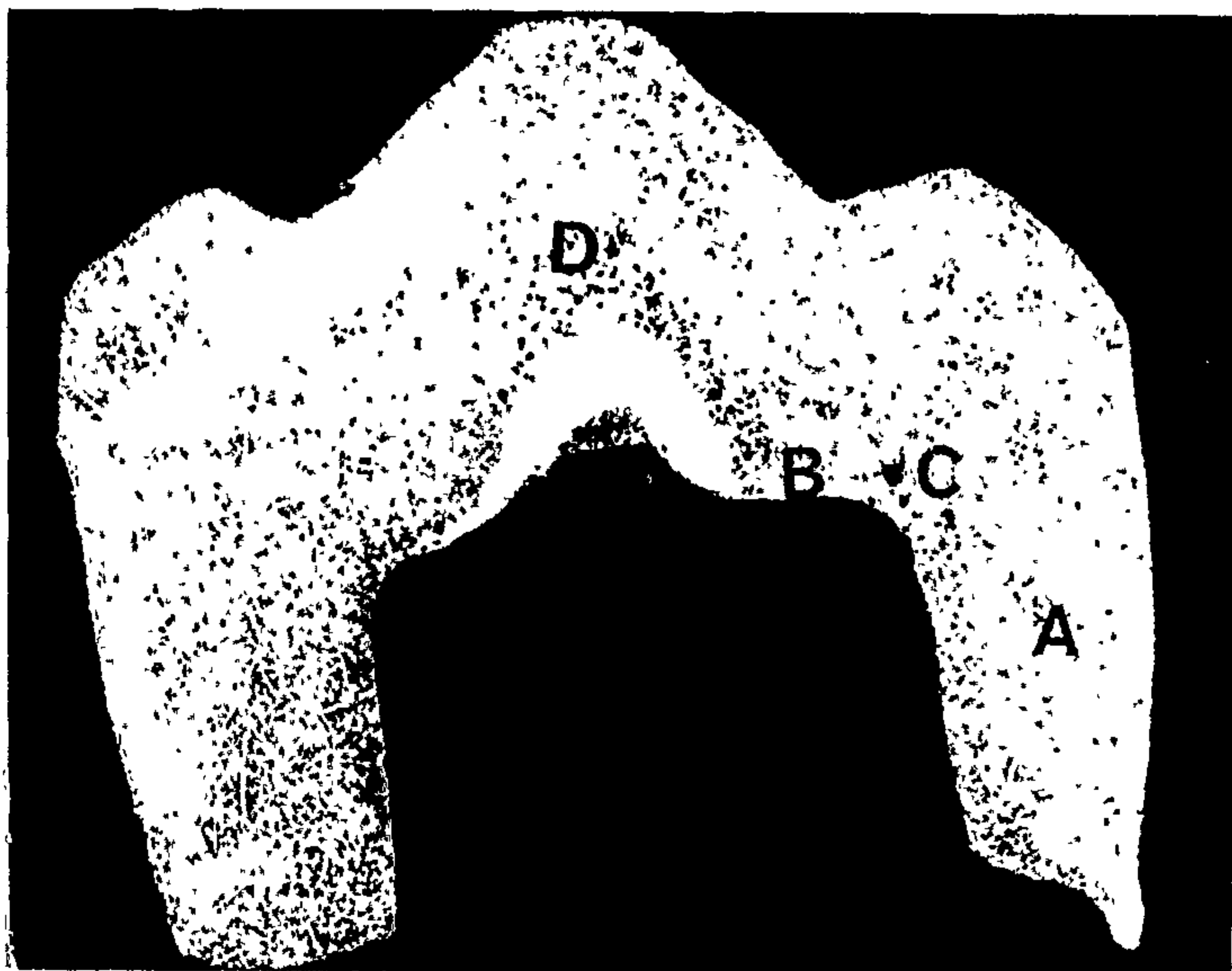
- A. Cast gold
- B. Electroformed gold matrix only present in the pulpal area
- C. Porosity due to gas inclusion
- D. Pinhole porosity (?).

Figure 17(x 10). Electrolytically etched.

Same specimen as figure 16. - (mirror image).

Legend.

- A. Cast gold (type C)
- B. Electroformed gold matrix only present in pulpal area
- C. Porosity due to gas inclusion
- D. Dendritic structure of porosity indicates microporosity.



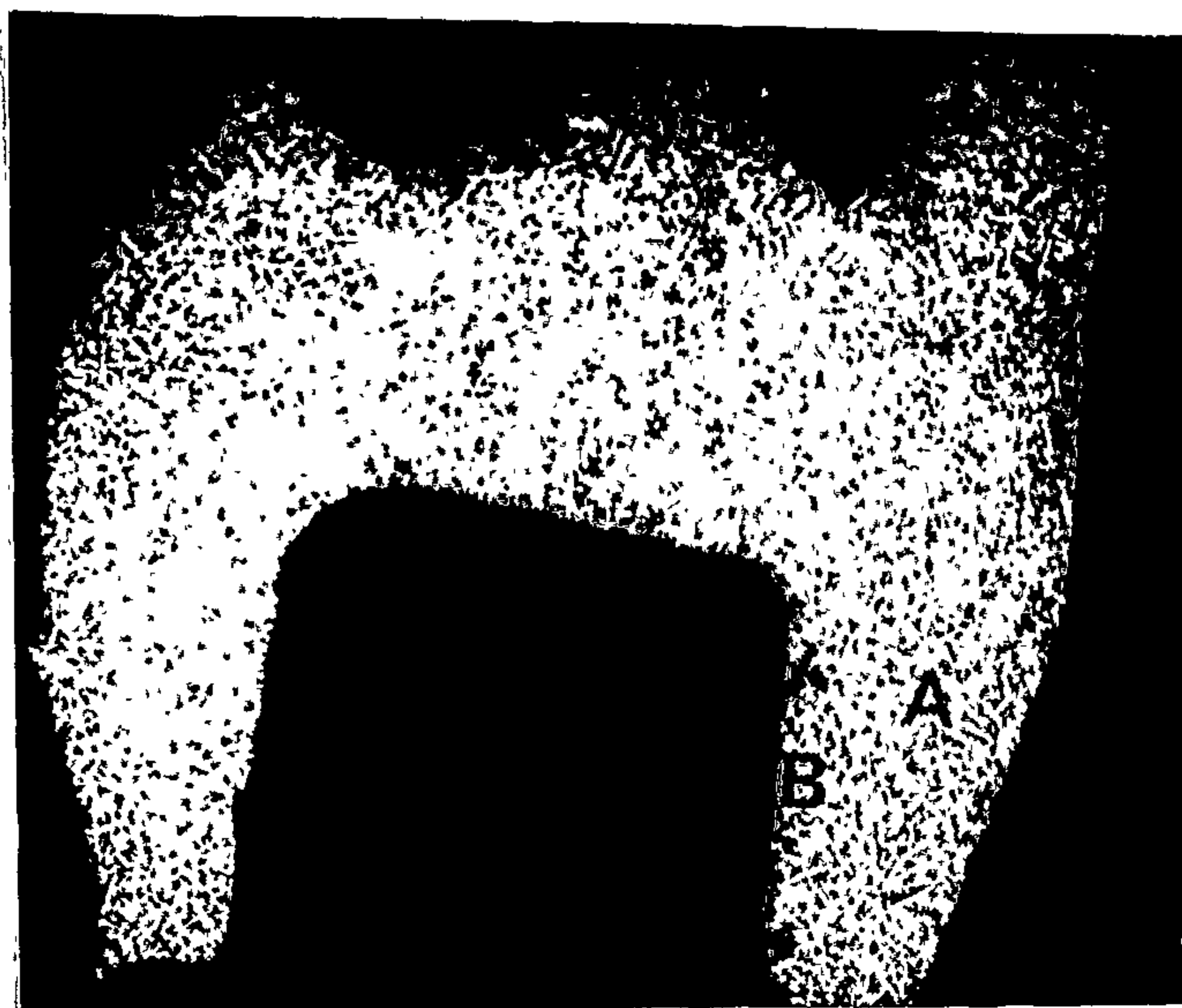


Figure 18(x 10).

Conditions.

Type C gold replaced in furnace at 700° for 24 hours after casting.

Legend.

A. Cast gold

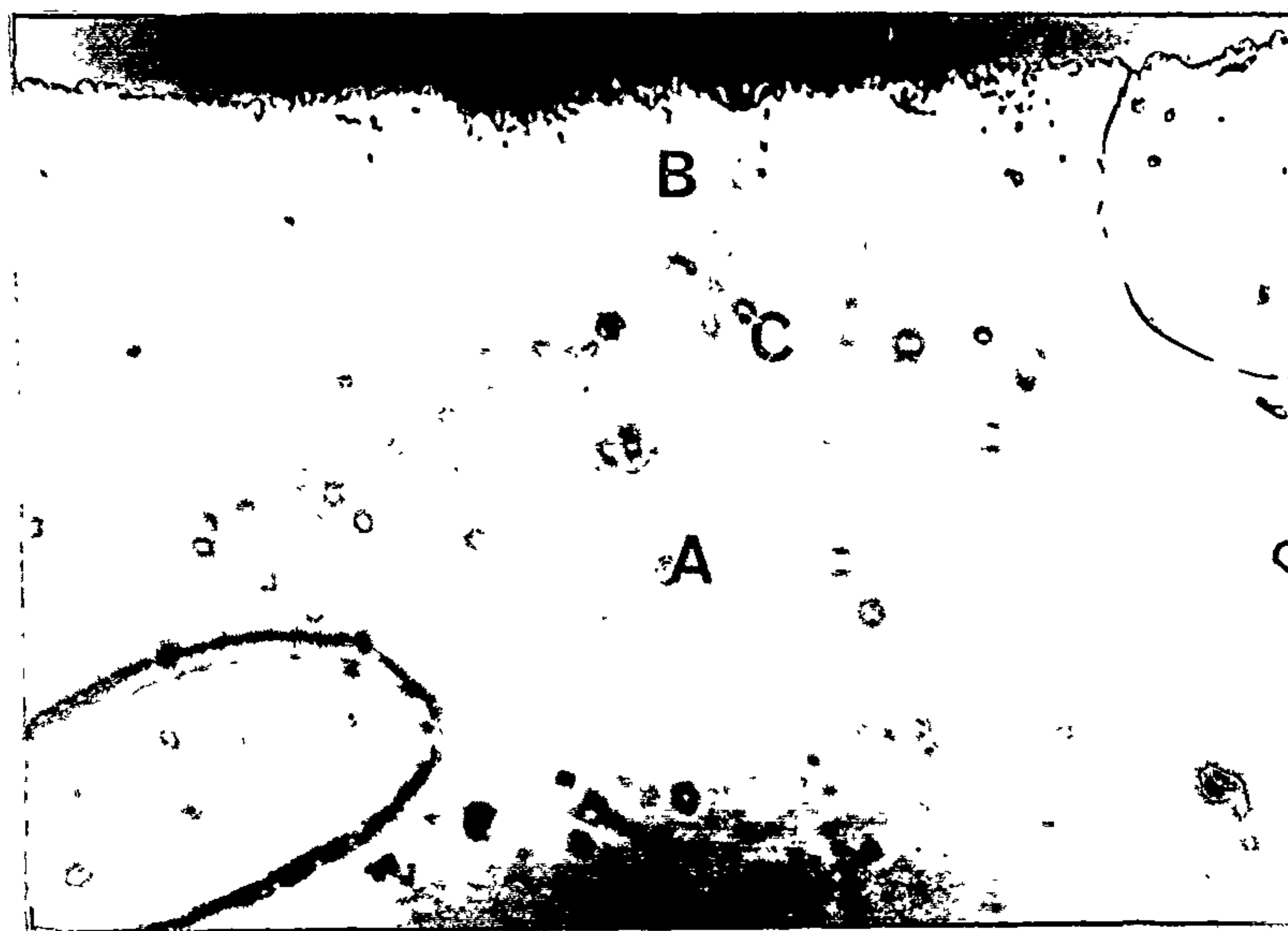
B. Matrix area.

Figure 19(x 400).

Higher magnification of the interface of figure 18.

Legend.

- A. Cast gold (type C)
- B. Matrix area
- C. Approximate position of interface between matrix
and cast gold.



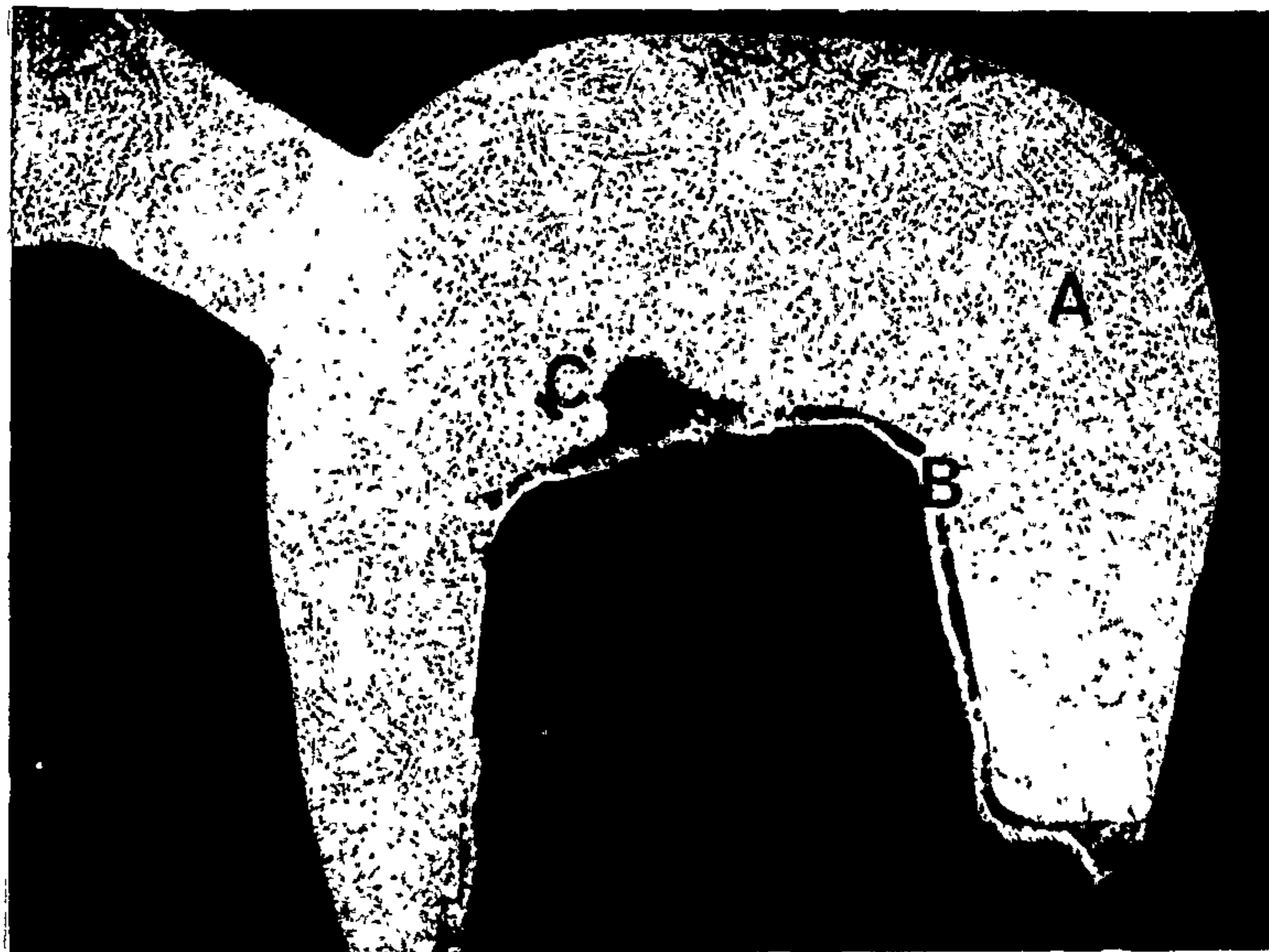


Figure 20(x 10).

Conditions.

Mould temperature 550°C

Casting temperature 955°C

Type B gold alloy.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Back pressure porosity.

Figure 21.(x 10).

Conditions.

Mould temperature 550°C

Casting temperature 985°C

Type B gold alloy.

Legend.

- A. Cast gold
- B. Electroformed gold matrix
- C. Porosity due to gas inclusion
- D. Pinhole porosity and microporosity.

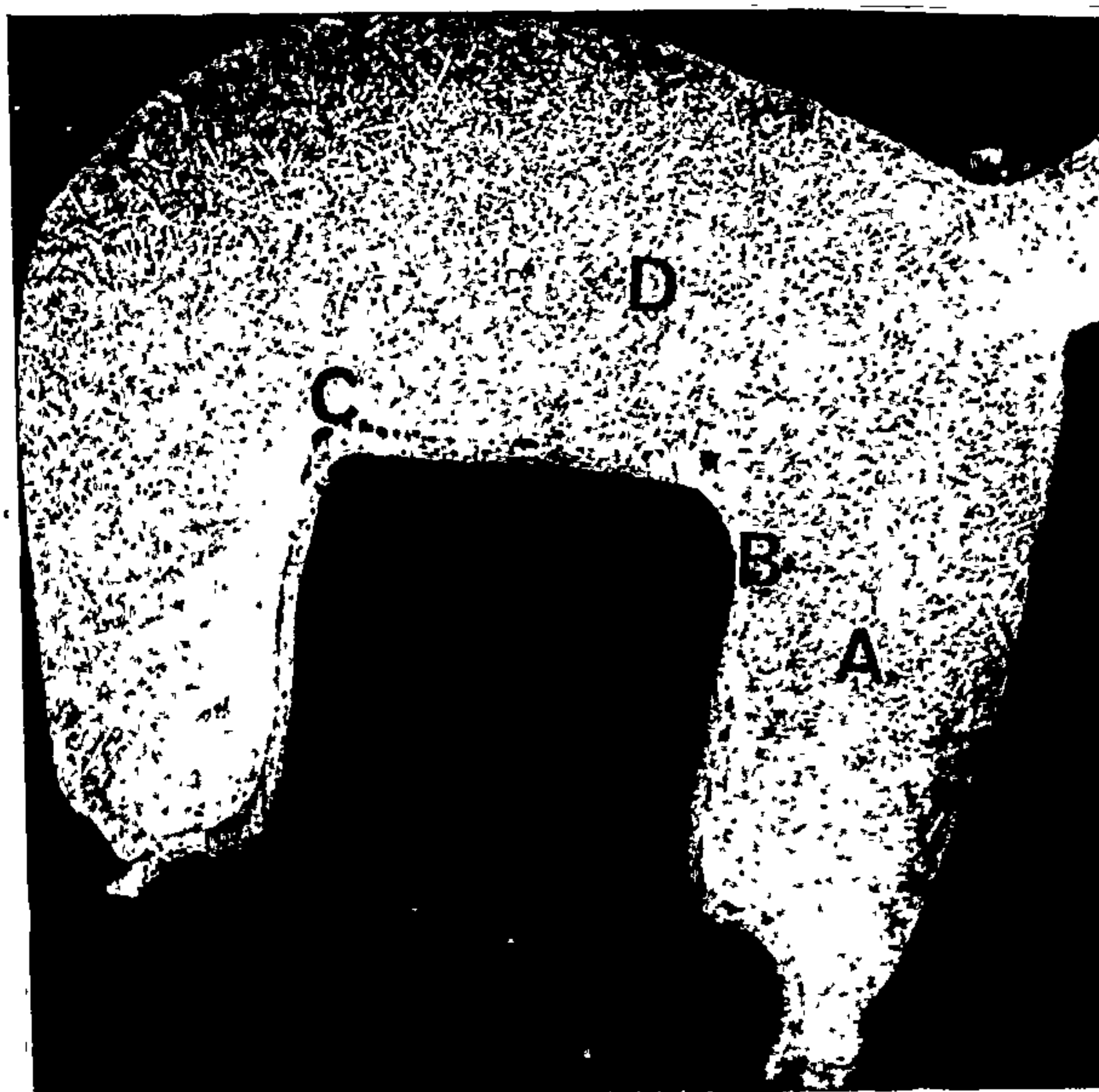




Figure 22(x 10).

Conditions.

Mould temperature 550°C

Casting temperature 980°C

Type C gold

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Back pressure porosity.

Figure 23(x 10).

Conditions.

Mould temperature 550°C

Casting temperature 1010°C

Type C gold.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Gaseous porosity

D. Area where freezing seems to have occurred independently from the bulk of the casting.

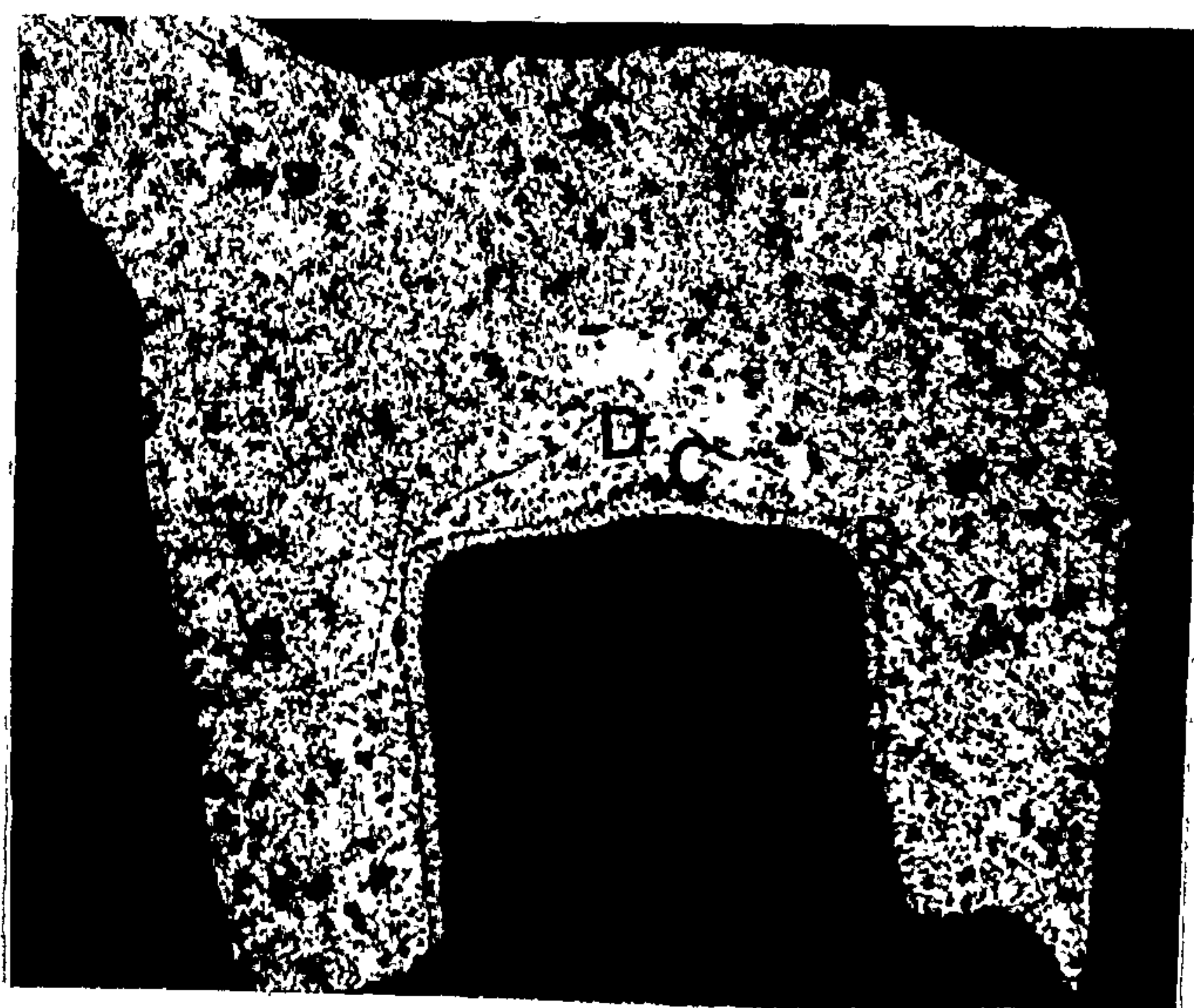




Figure 24(x 10).

Conditions.

Mould temperature 650°C

Casting temperature 955°C

Type Bⁱ gold alloy.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to gas inclusion.

Figure 25(x 10).

Conditions.

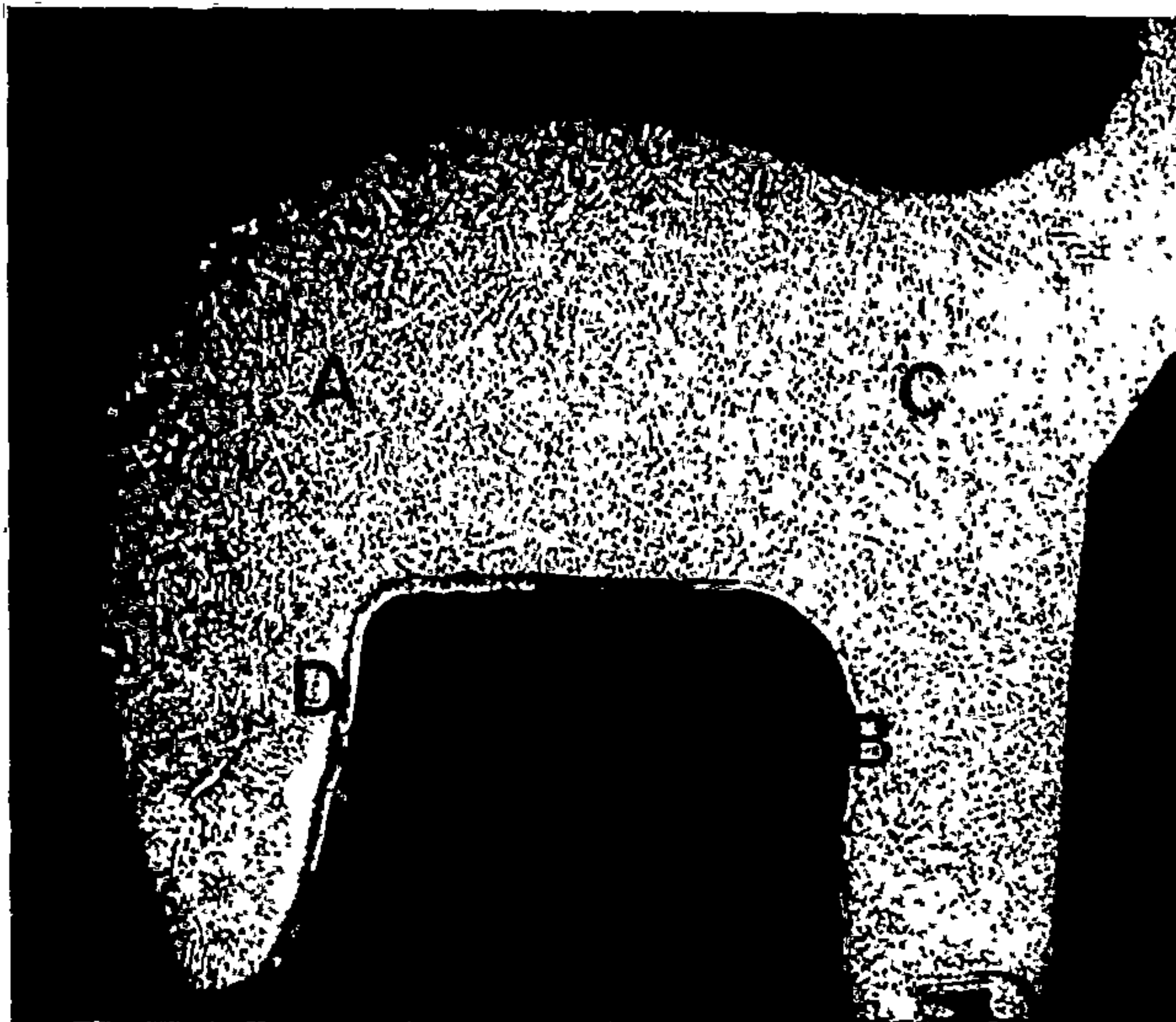
Mould temperature 650°C

Casting temperature 985°C

Type Bⁱ gold alloy.

Legend.

- A. Cast gold
- B. Electroformed gold matrix
- C. Dendritic shrinkage - localised shrinkage porosity
- D. Matrix stripped away from casting.



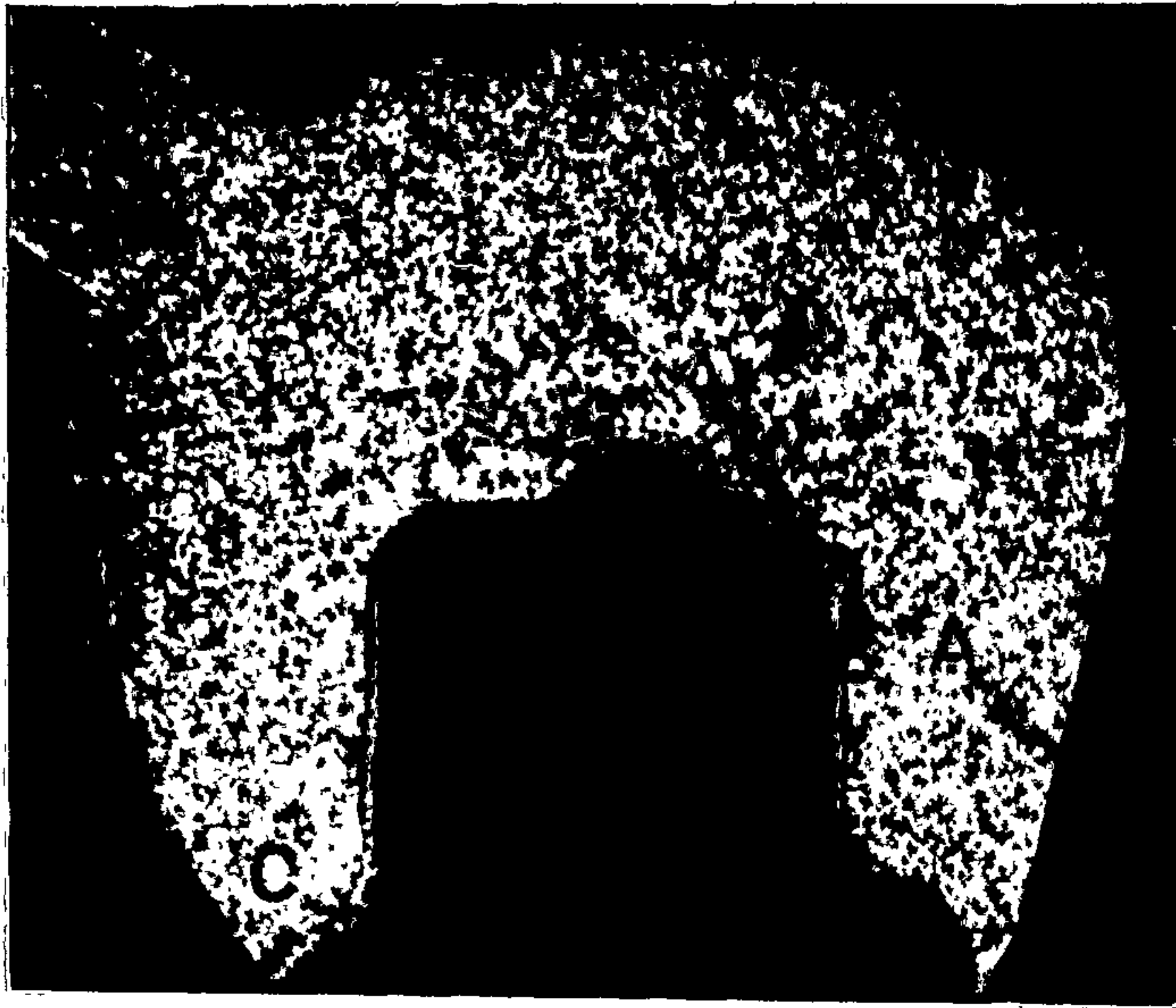


Figure 26(x 10).

Conditions.

Mould temperature 650°C

Casting temperature 980°C

Type C gold alloy.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to gas inclusion.

Figure 27(x 10).Conditions.

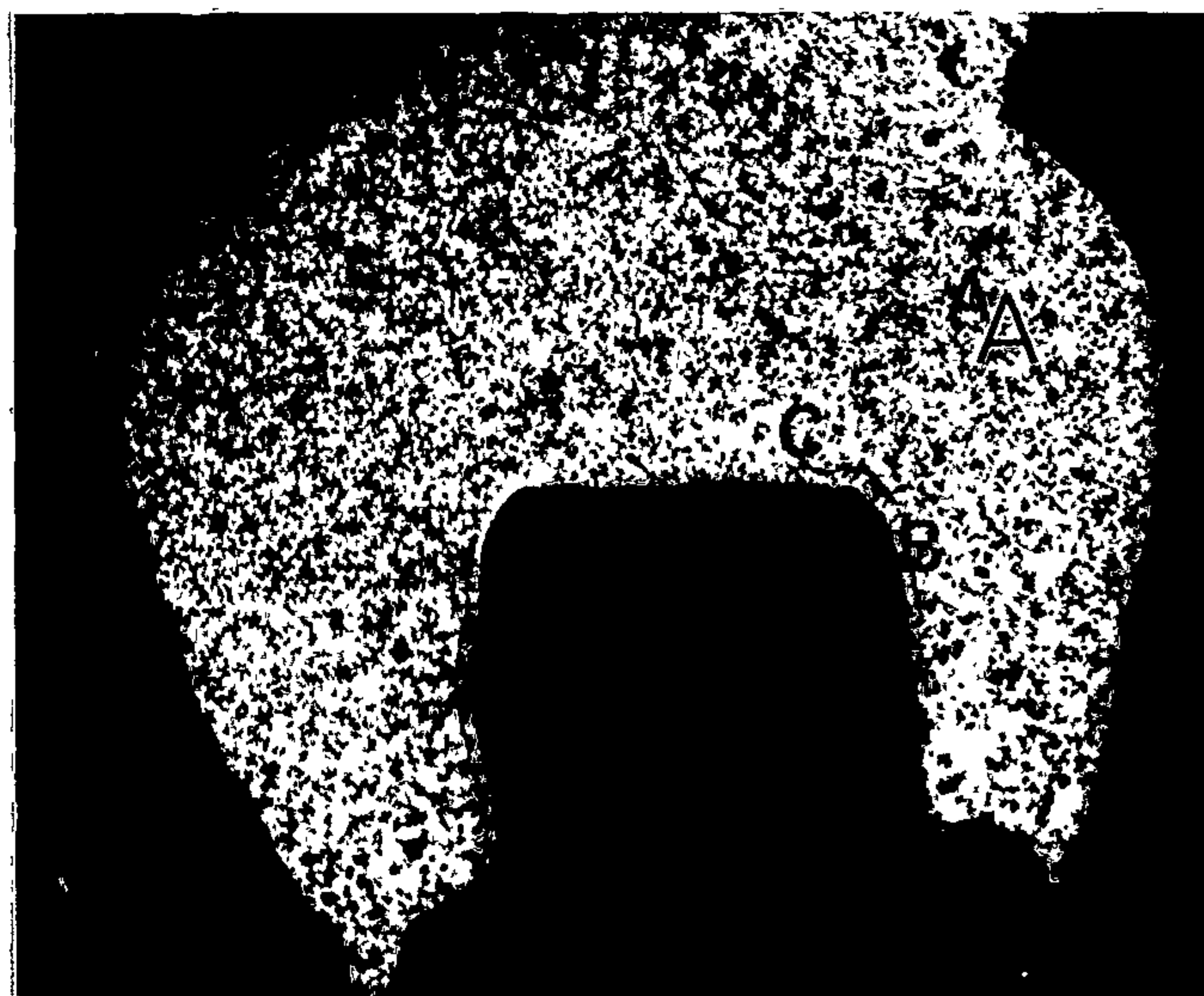
Mould temperature 650°C

Casting temperature 1010°C

Type C gold alloy.

Legend.

- A. Cast gold
- B. Electroformed gold matrix
- C. Porosity due to gas inclusion.



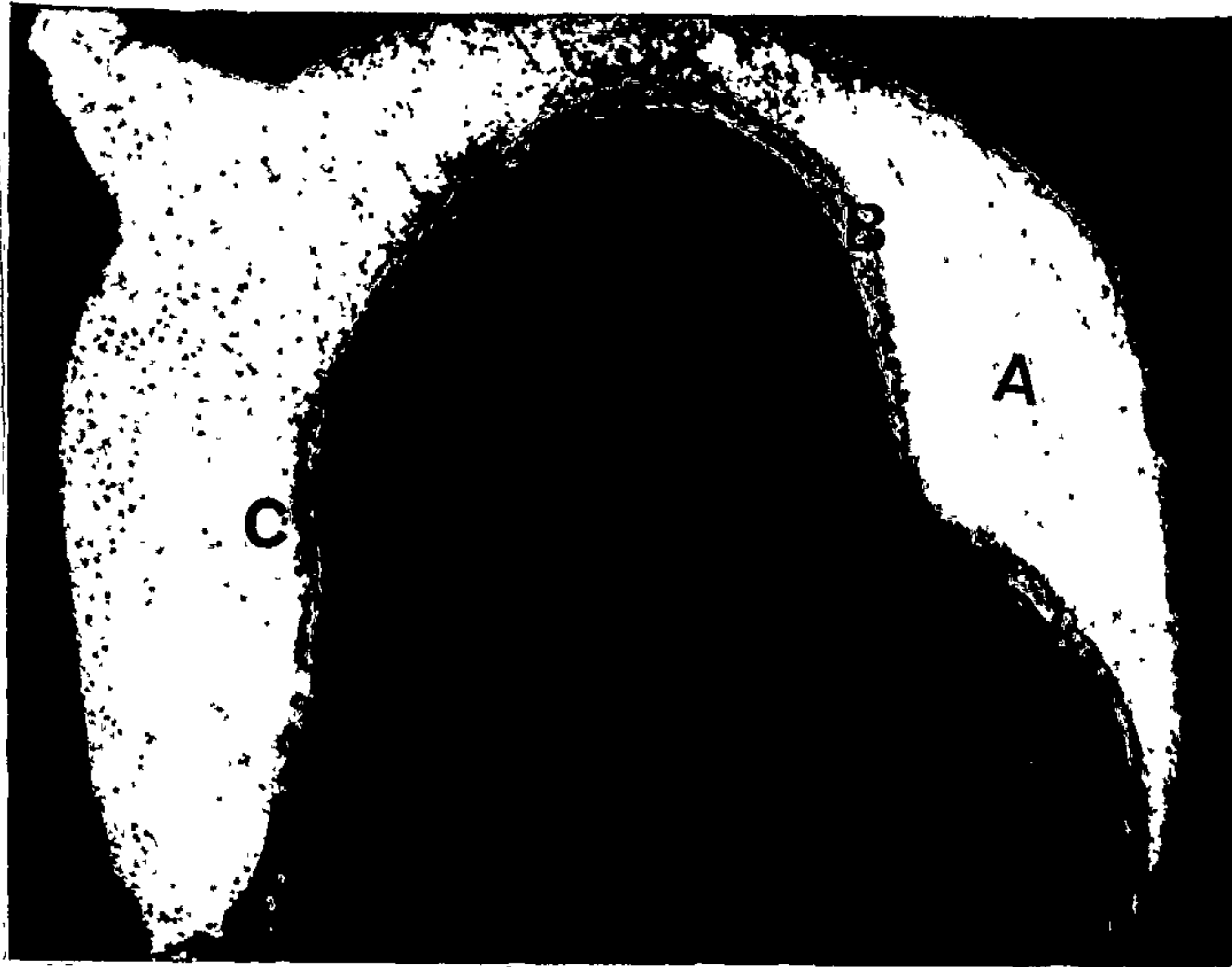


Figure 28(x 10).

Conditions.

Mould temperature 750°C

Casting temperature 955°C

Type Bⁱ gold alloy.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to gas inclusion.

Figure 29(x 10).

Conditions.

Mould temperature 750°C

Casting temperature 980°C

Type Bⁱ gold alloy.

Legend.

- A. Cast gold
- B. Electroformed gold matrix
- C. Subsurface porosity
- D. Porosity due to gas inclusion.



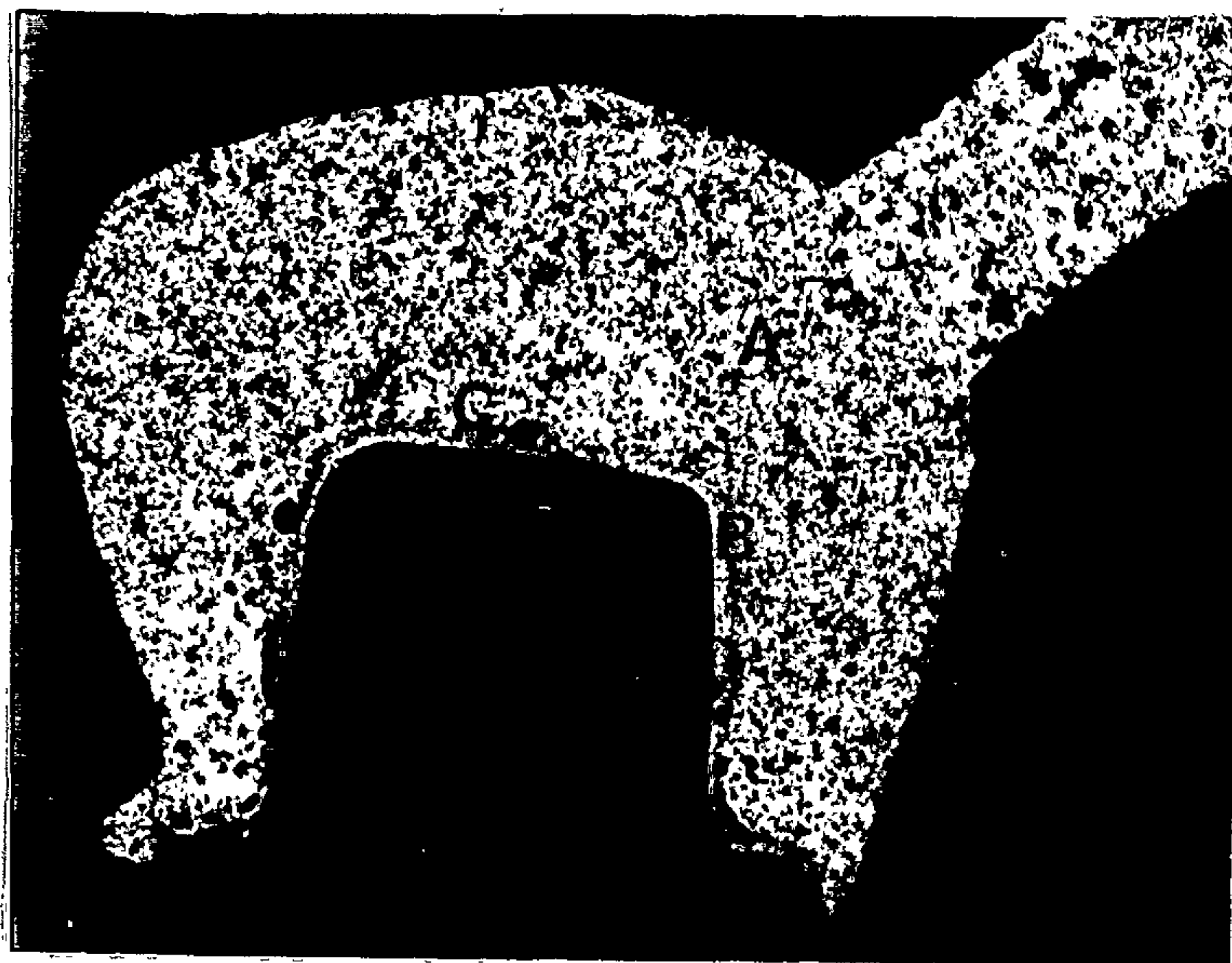


Figure 30(x 10).

Conditions.

Mould temperature 750°C

Casting temperature 980°C

Type C gold alloy.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to gas inclusion.

Figure 31(x 10).

Conditions.

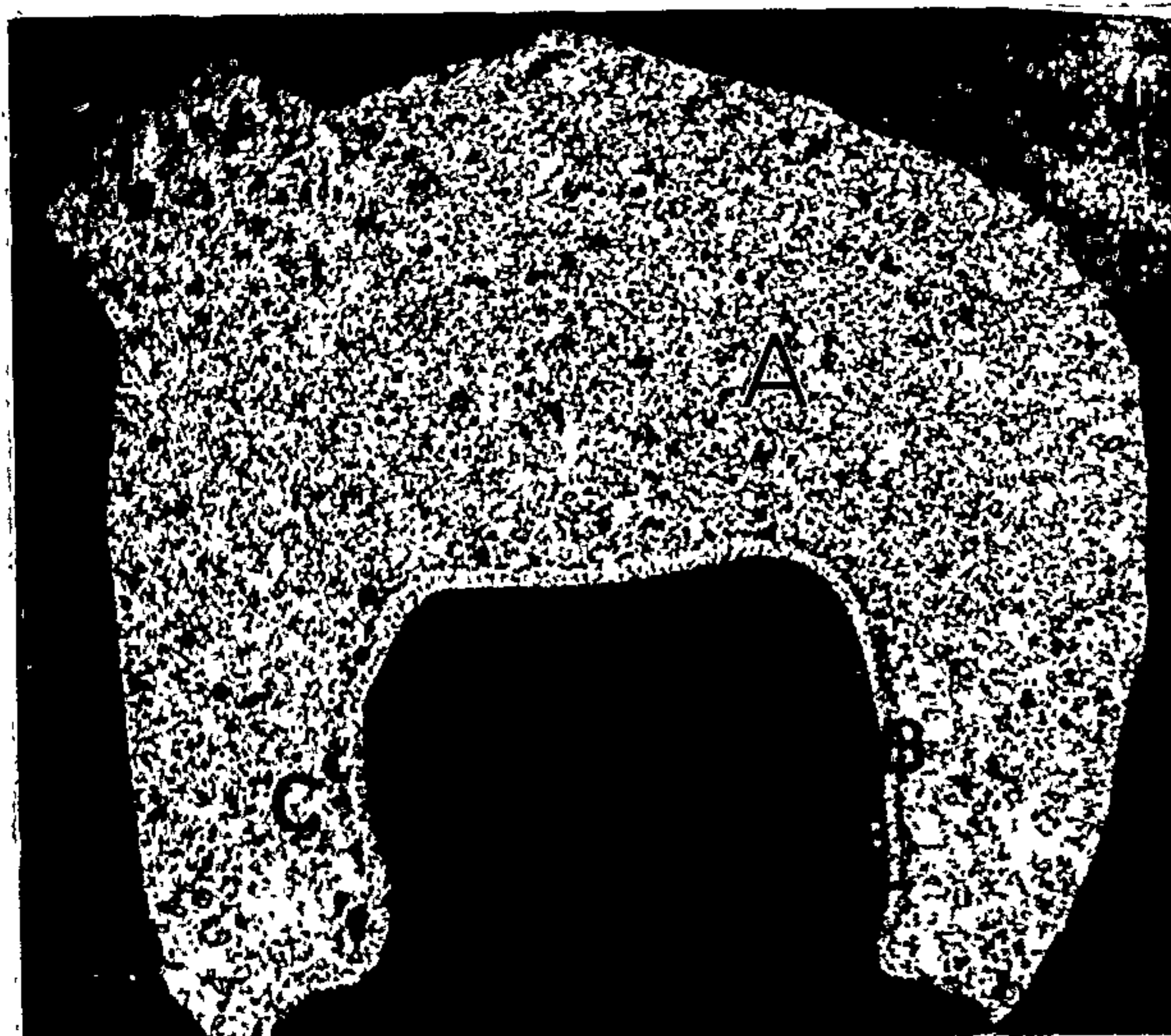
Mould temperature 750°C

Casting temperature 1010°C

Type C gold alloy.

Legend.

- A. Cast gold
- B. Electroformed gold matrix
- C. Porosity due to gas inclusion.



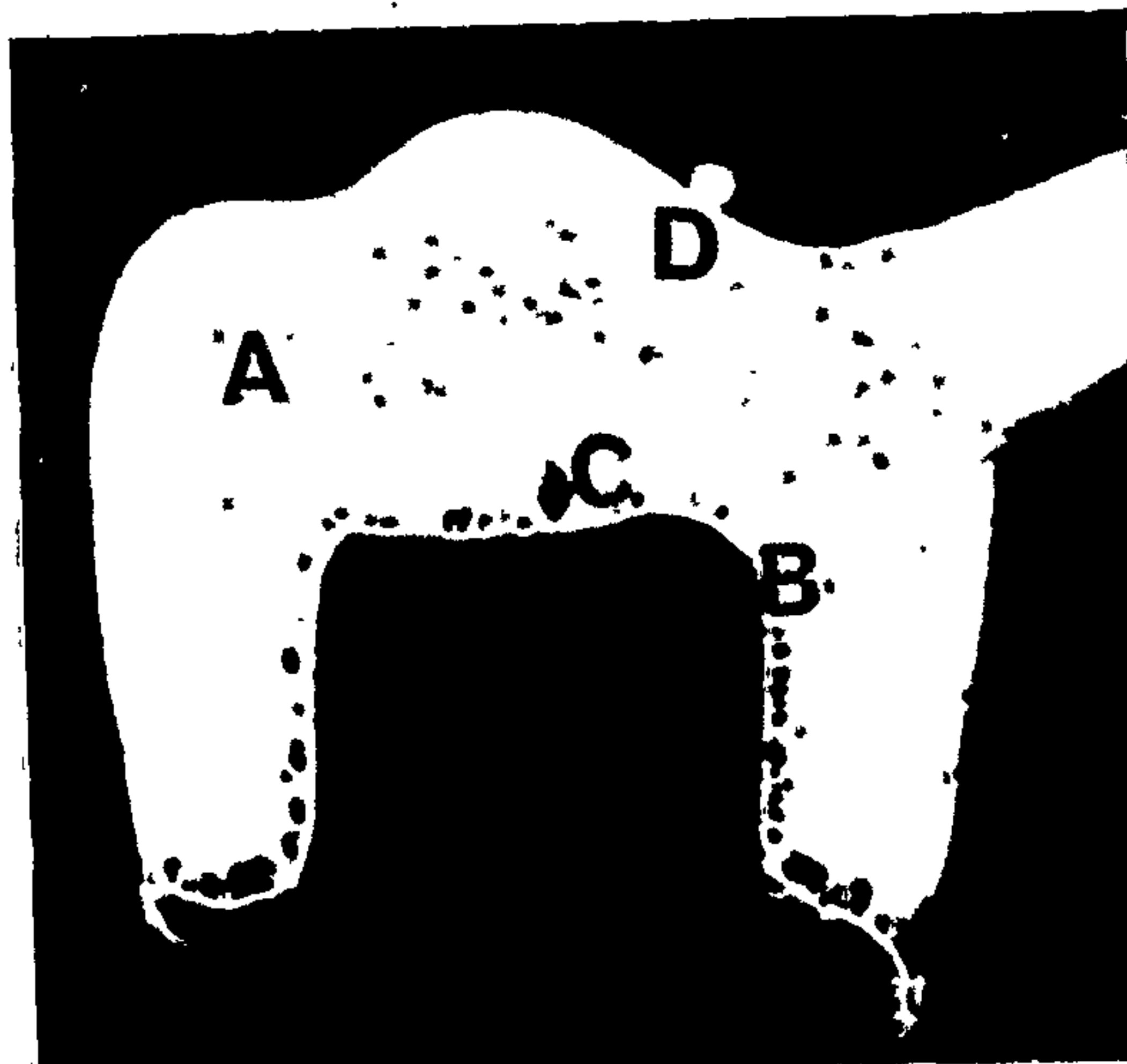


Figure 32(x 8). Etched in aqua regia.

Conditions.

Mould temperature 750°C

Casting temperature 1015°C

Type Bⁱⁱ gold alloy.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to gas inclusion

D. Surface nodule.

Figure 33(x 8). Etched in aqua regia.

Conditions.

Mould temperature 750°C

Casting temperature 1015°C

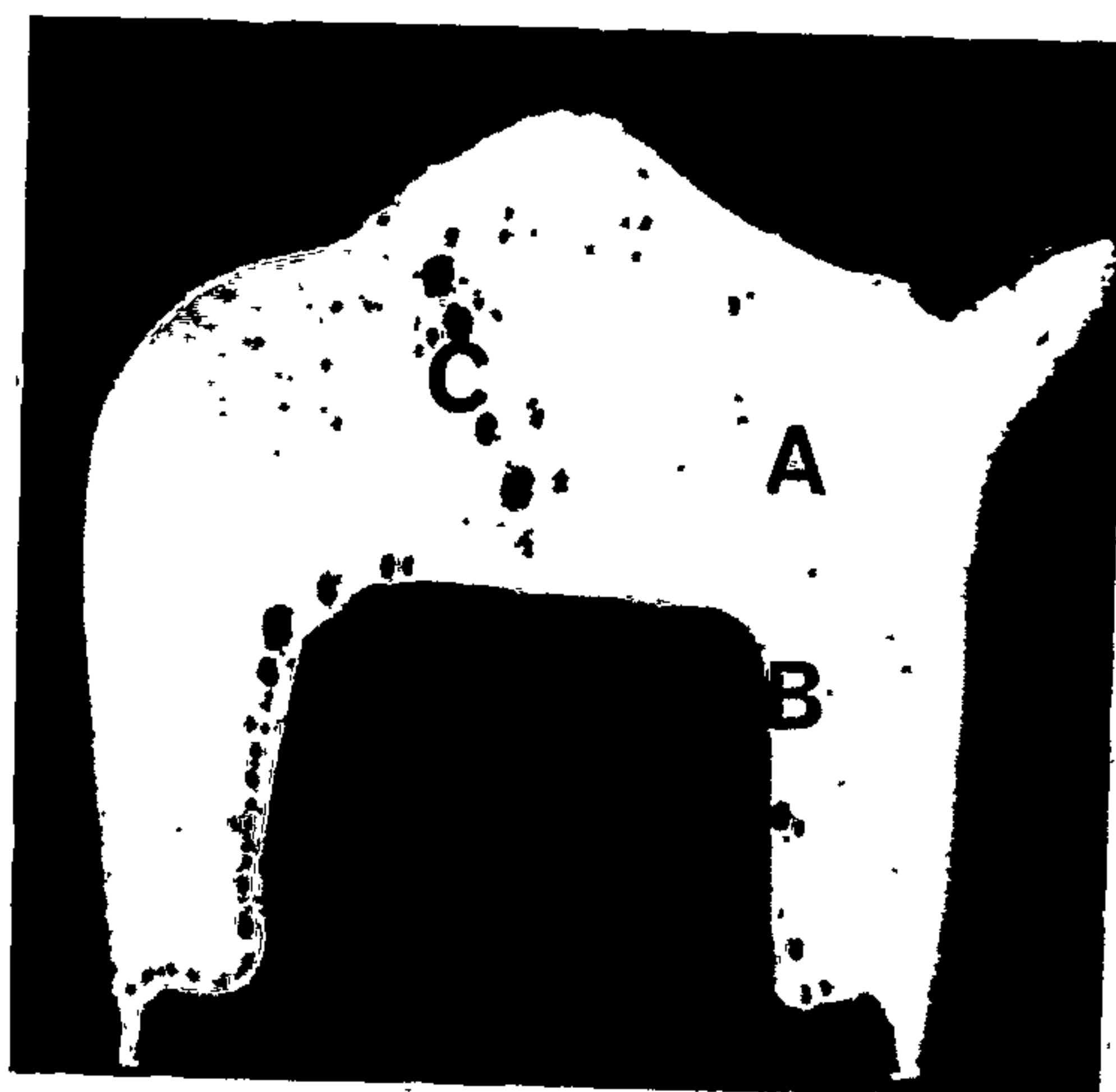
Type Bⁱⁱ gold alloy.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to gas inclusion.



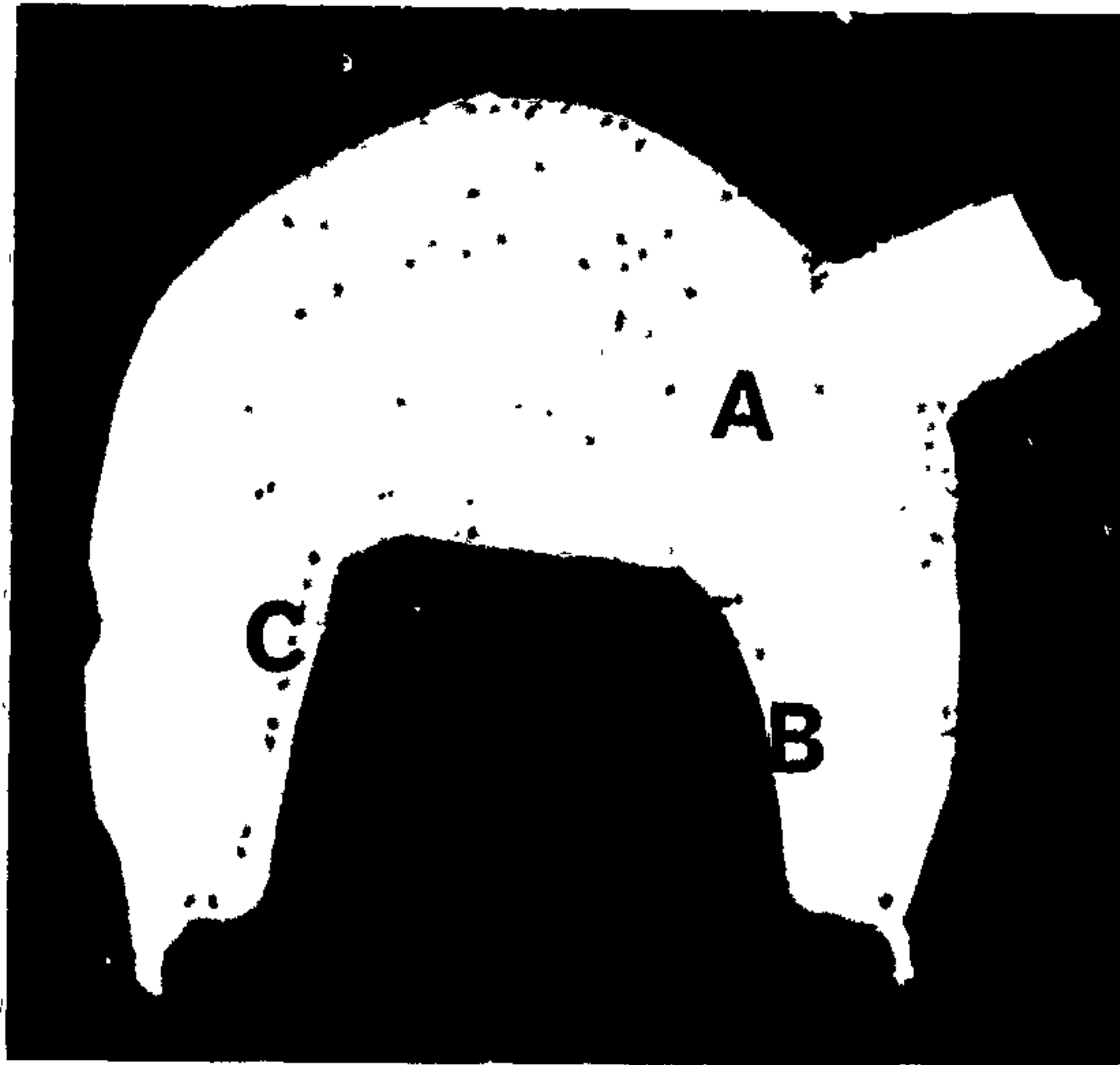


Figure 34(x 8). Etched in aqua regia.

Conditions.

Mould temperature 750°C

Casting temperature 960°C

Type D gold alloy.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to gas inclusion.

Figure 35(x 8). Etched in aqua regia.

Conditions.

Mould temperature 750°C

Casting temperature 960°C

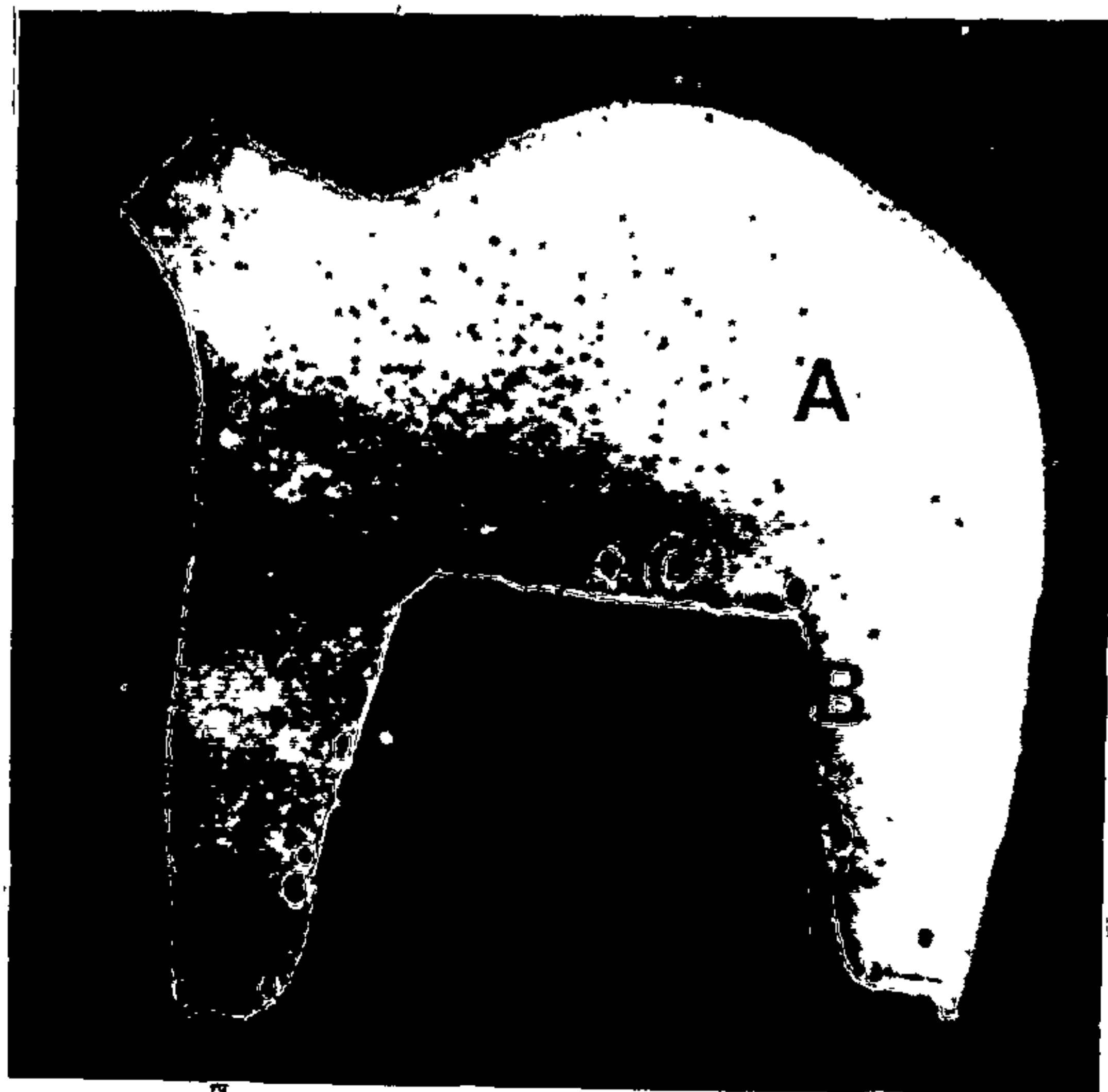
Type D gold alloy.

Legend.

A. Cast gold

B. Electroformed gold matrix

C. Porosity due to gas inclusion.



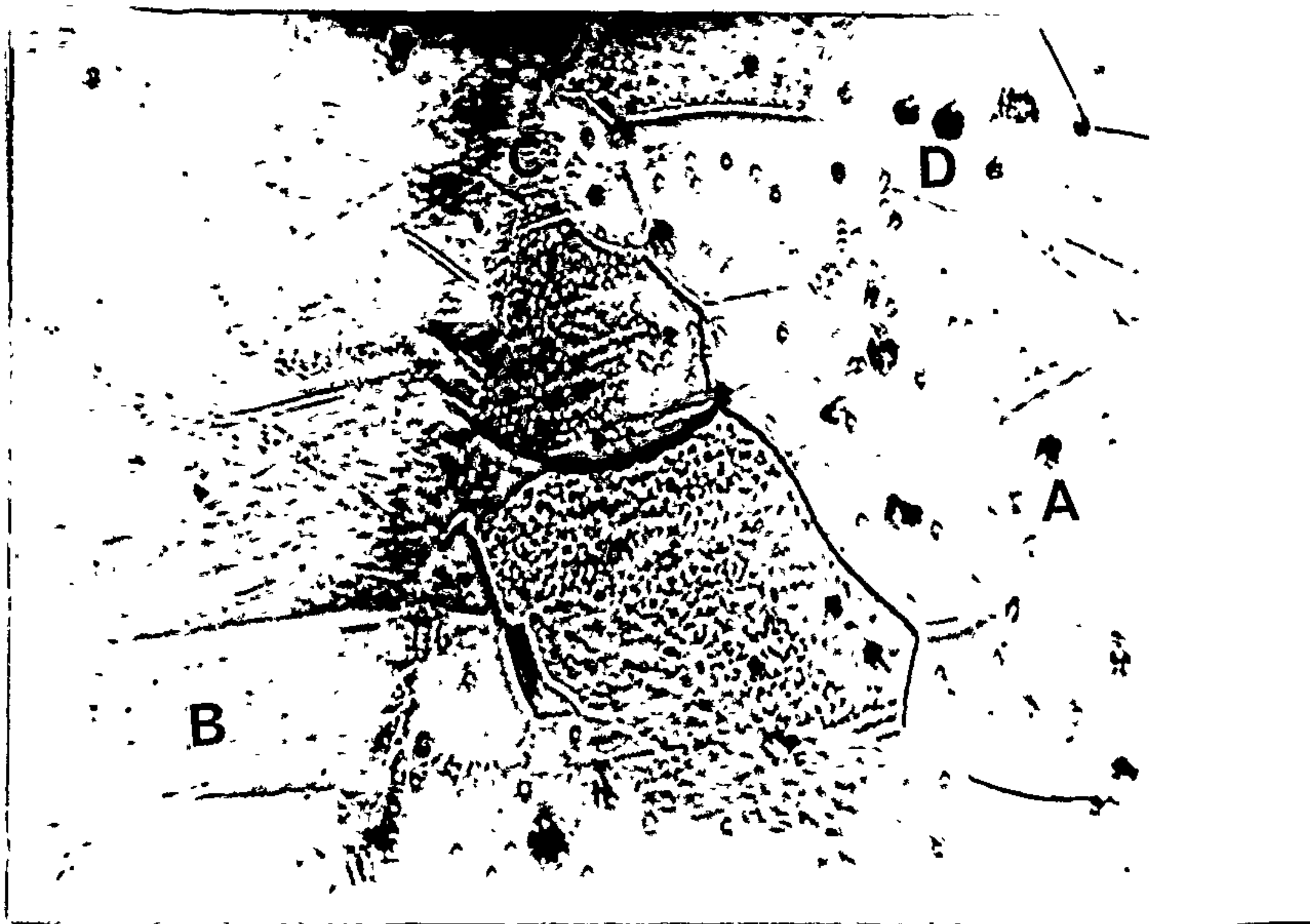


Figure 36. Electroformed gold - gold alloy interface (x 400).

Oblique lighting, showing tight union at interface.

(Same specimen as shown in Figure 25.)

Legend.

- A. Cast gold alloy (Type Bⁱ)
- B. Electroformed gold matrix
- C. Interface
- D. Pinhole porosity.

Figure 37. Electroformed gold - gold alloy interface (x 400).

(Same specimen as shown in Figure 25.)

Legend.

- A. Cast gold alloy (Type Bⁱ)
- B. Electroformed gold matrix
- C. Interface
- D. Pinhole porosity
- E. Merging of grain boundaries at interface.



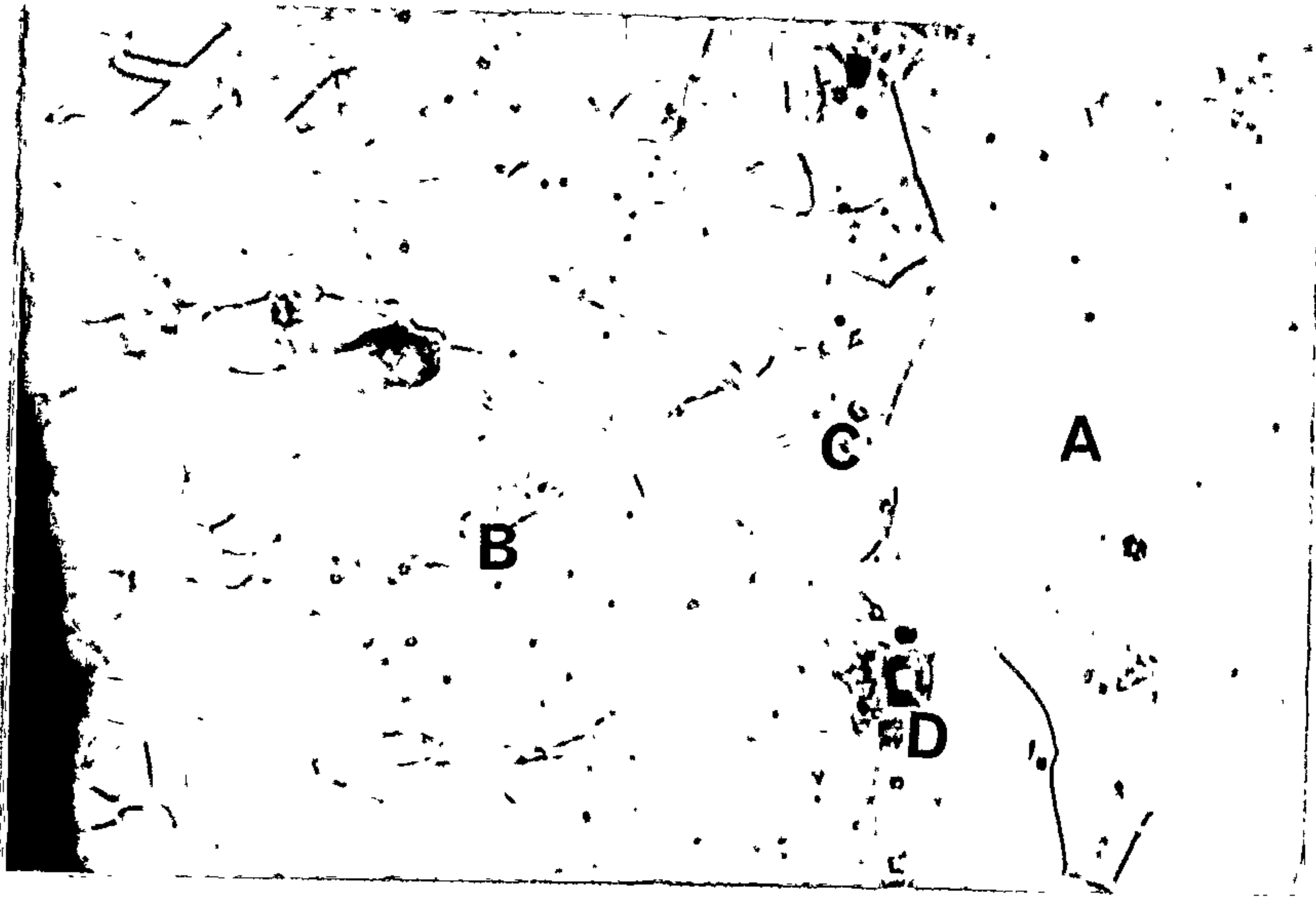


Figure 38. Electroformed gold - gold alloy interface (x 400).

(Same specimen as shown in Figure 27.)

Legend.

- A. Cast gold alloy (Type C)
- B. Electroformed gold matrix
- C. Interface showing diffusion
- D. Inclusion.

Figure 39. Electroformed gold - gold alloy interface (x 400).

(Same specimen as shown in Figure 31.)

Legend.

- A. Cast gold alloy (Type C)
- B. Electroformed gold matrix
- C. Interface showing diffusion
- D. Pinhole porosity.

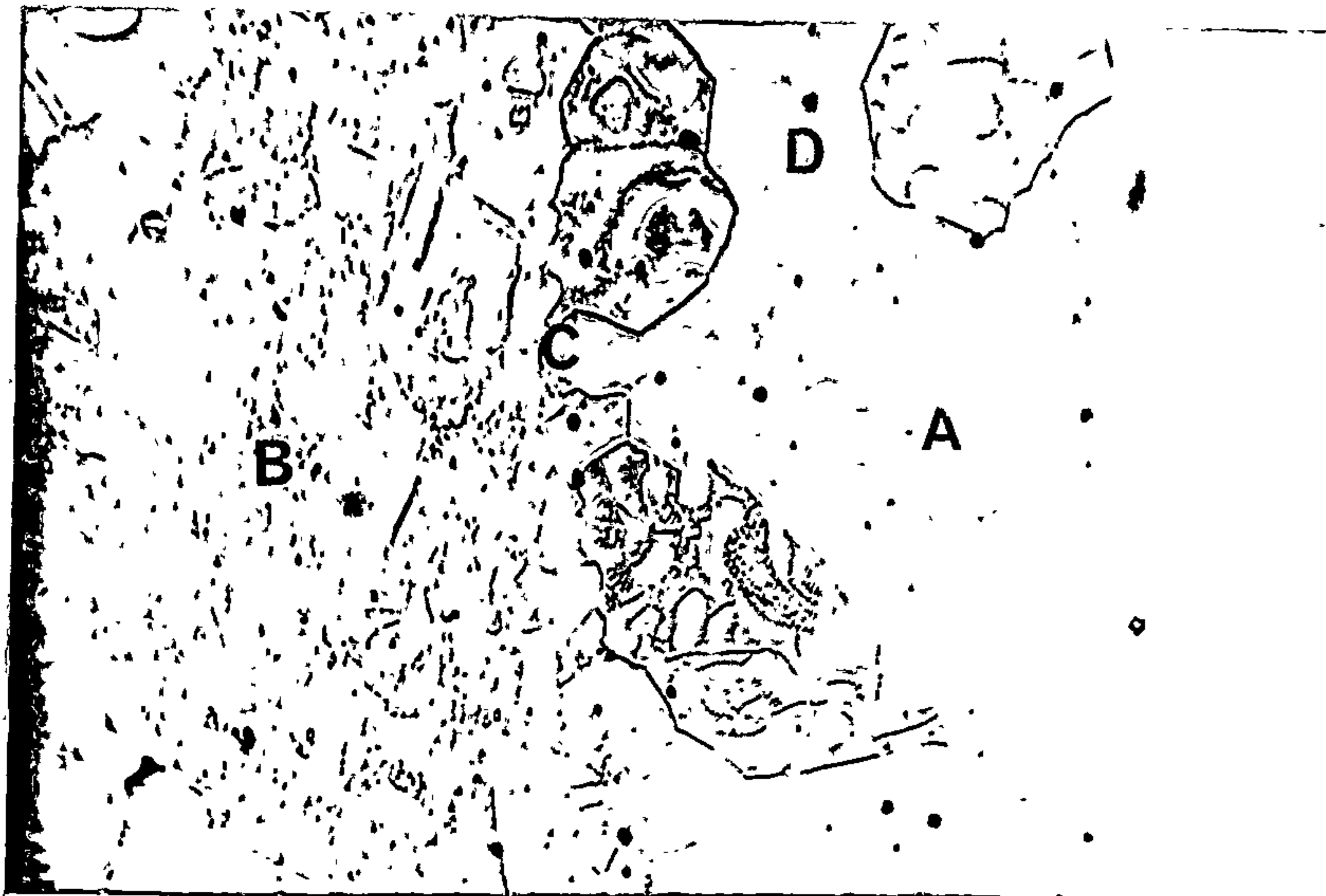




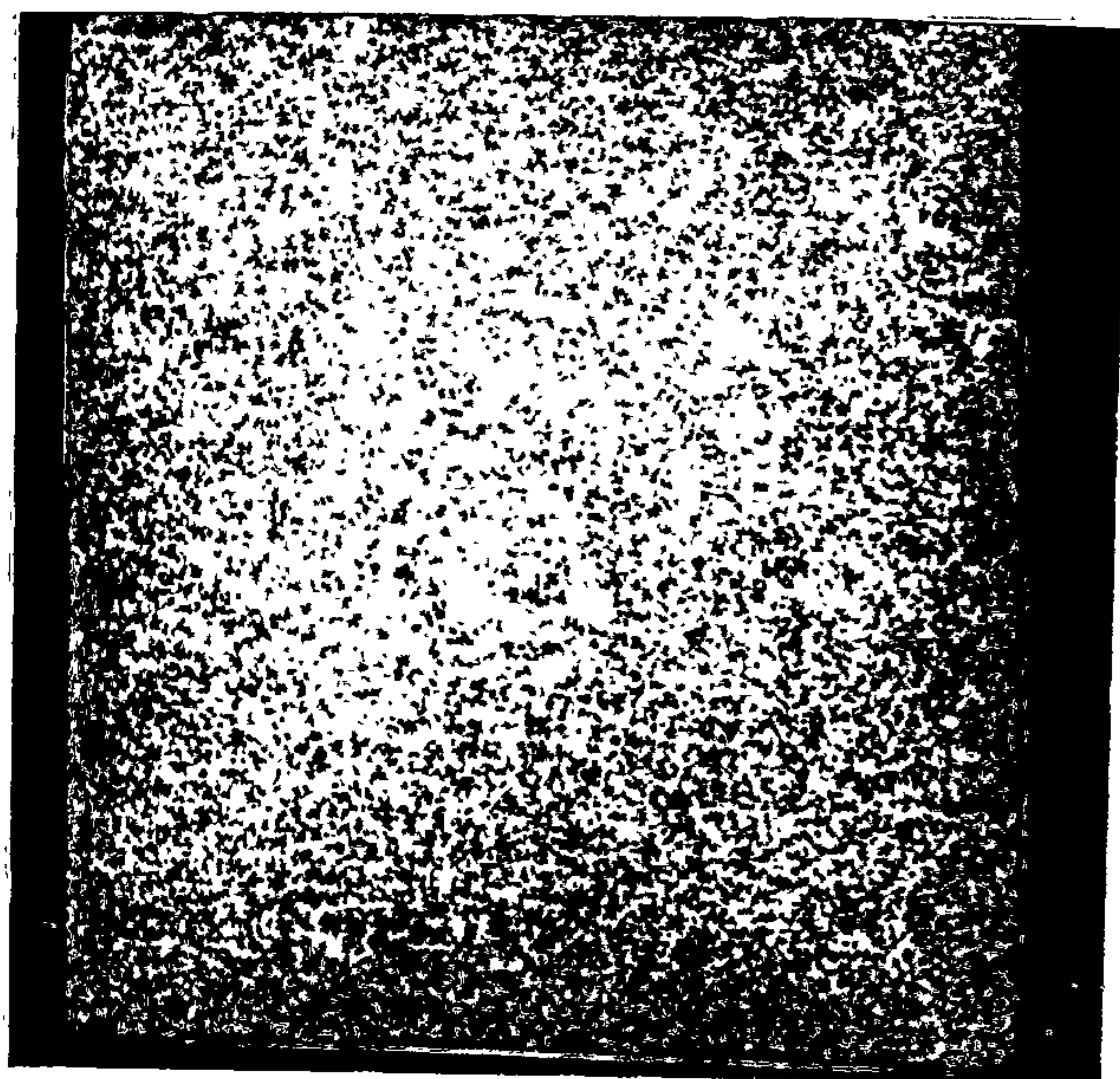
Figure 40 (x 350). Knoop and Vickers hardness testing 100g. load.

Legend.

- A. Cast gold alloy (Type C)
- B. Electroformed gold matrix
- C. Interface
- D. Pinhole porosity.

Figure 41(x 15).

Rolled type C alloy, as supplied by the manufacturer,
showing fine grain structure.



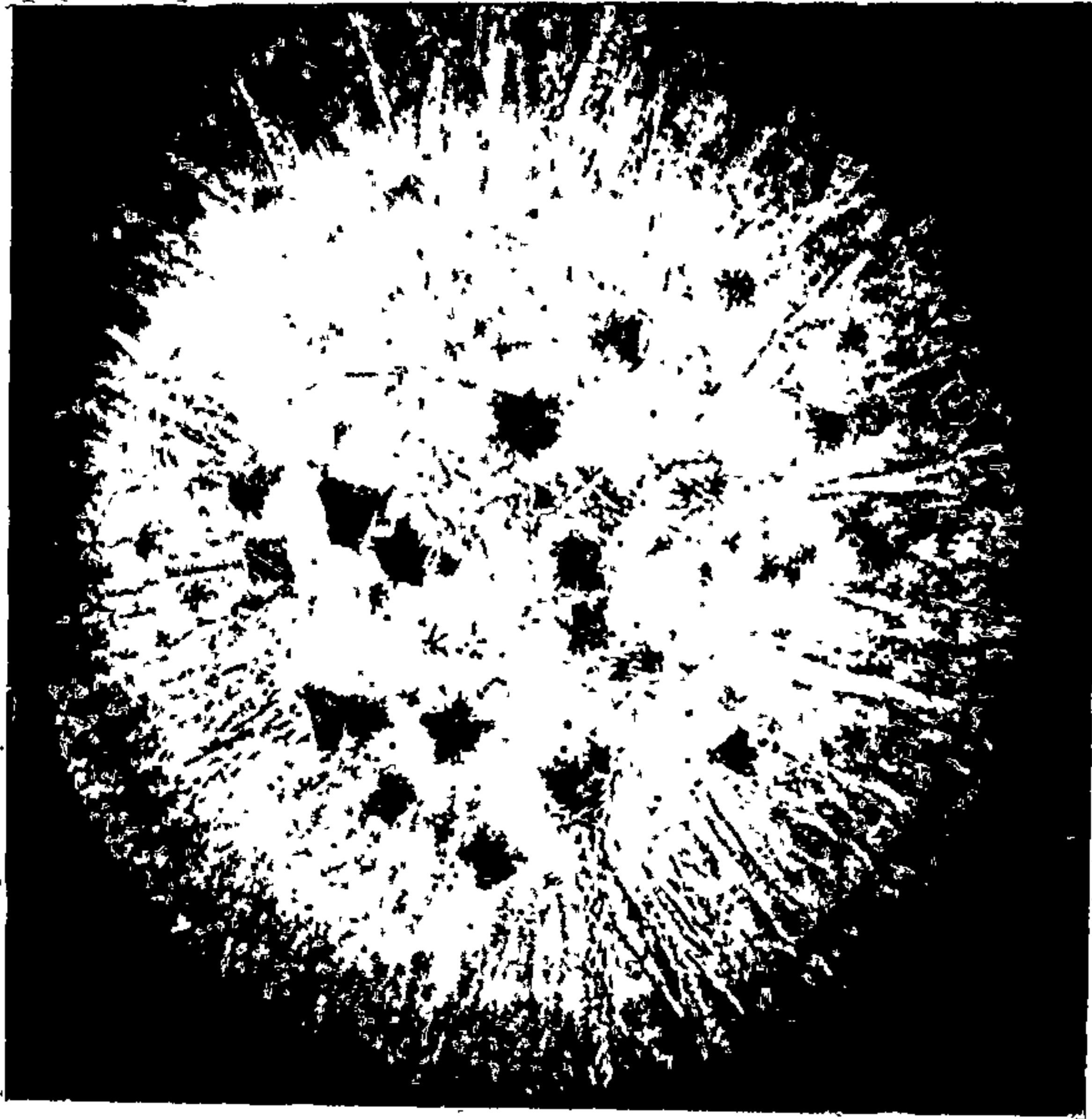
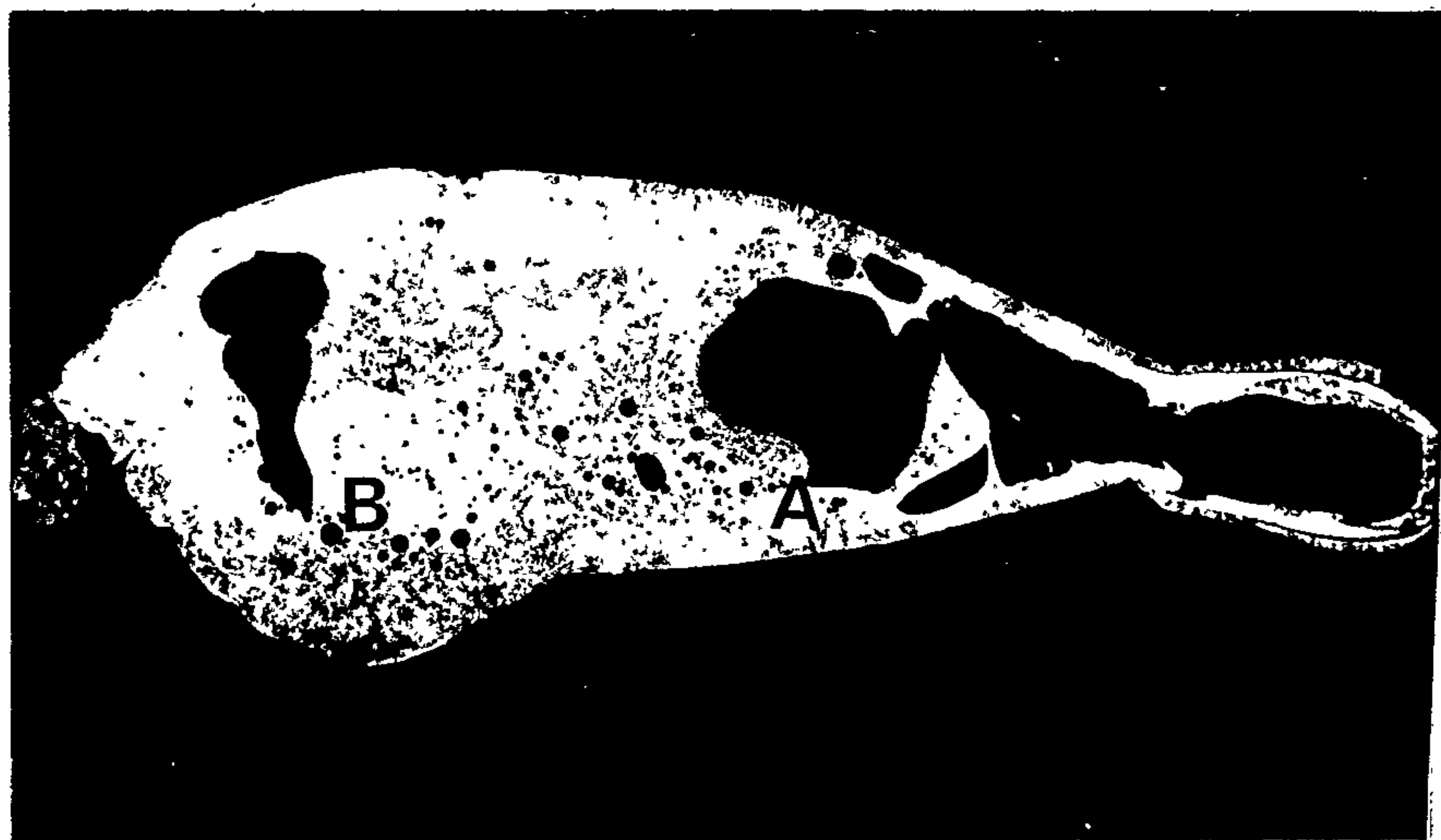


Figure 42(x 15).

Granulated type Bⁱ alloy, as supplied by the manufacturer,
showing a radially grained structure.

Figure 43(x 15).

Granulated type Bⁱ gold alloy, as supplied by the manufacturer, showing large(A) and small areas(B) of occluded gas.



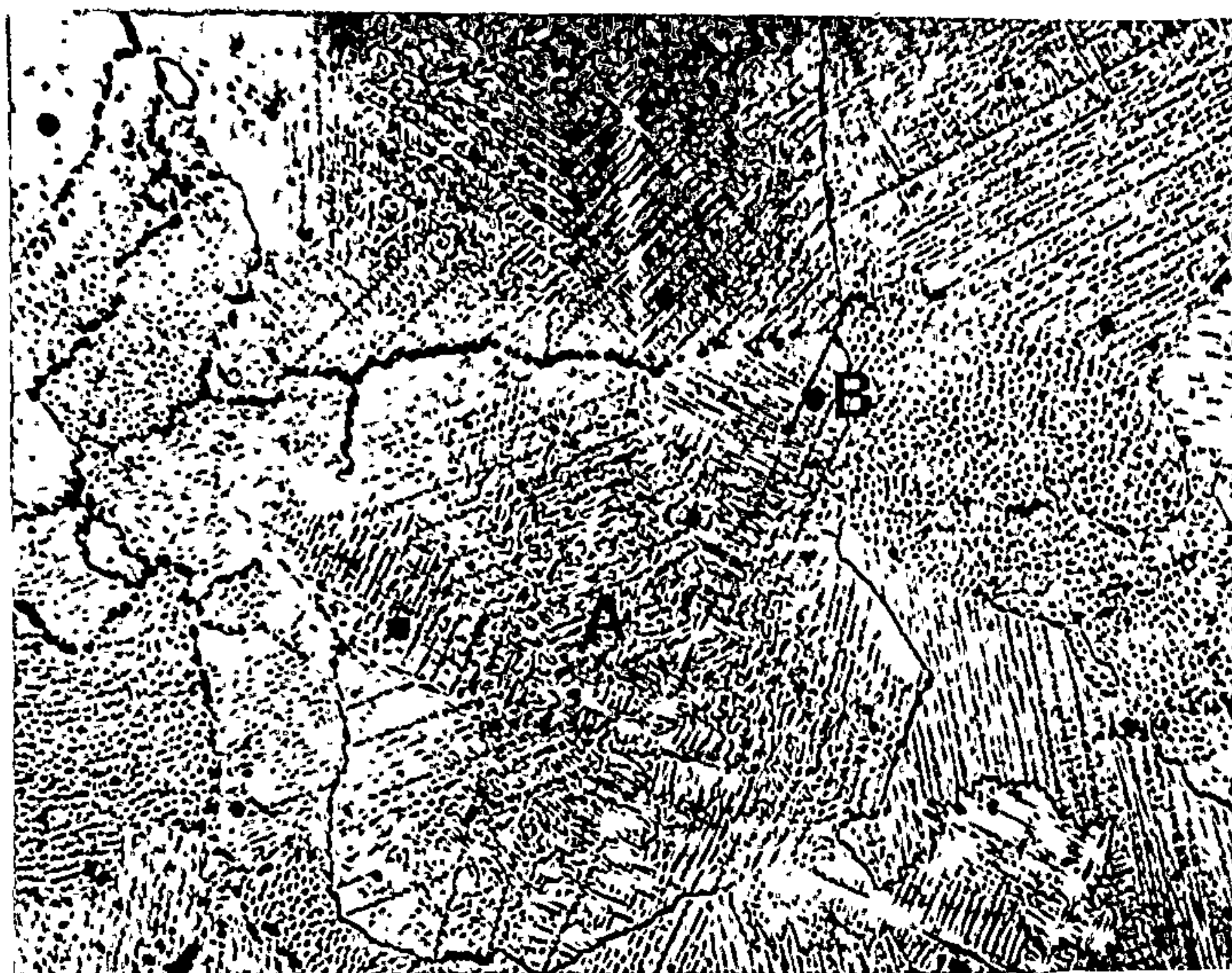


Figure 44. Type Bⁱ gold alloy (x 200).

Legend.

- A. Grain showing dendritic structure
- B. Pinhole porosity.

Figure 45. Type Bⁱ gold alloy, (x 350).

Legend.

A. Dendritic shrinkage showing ragged, labyrinthine appearance

B. Gaseous porosity.

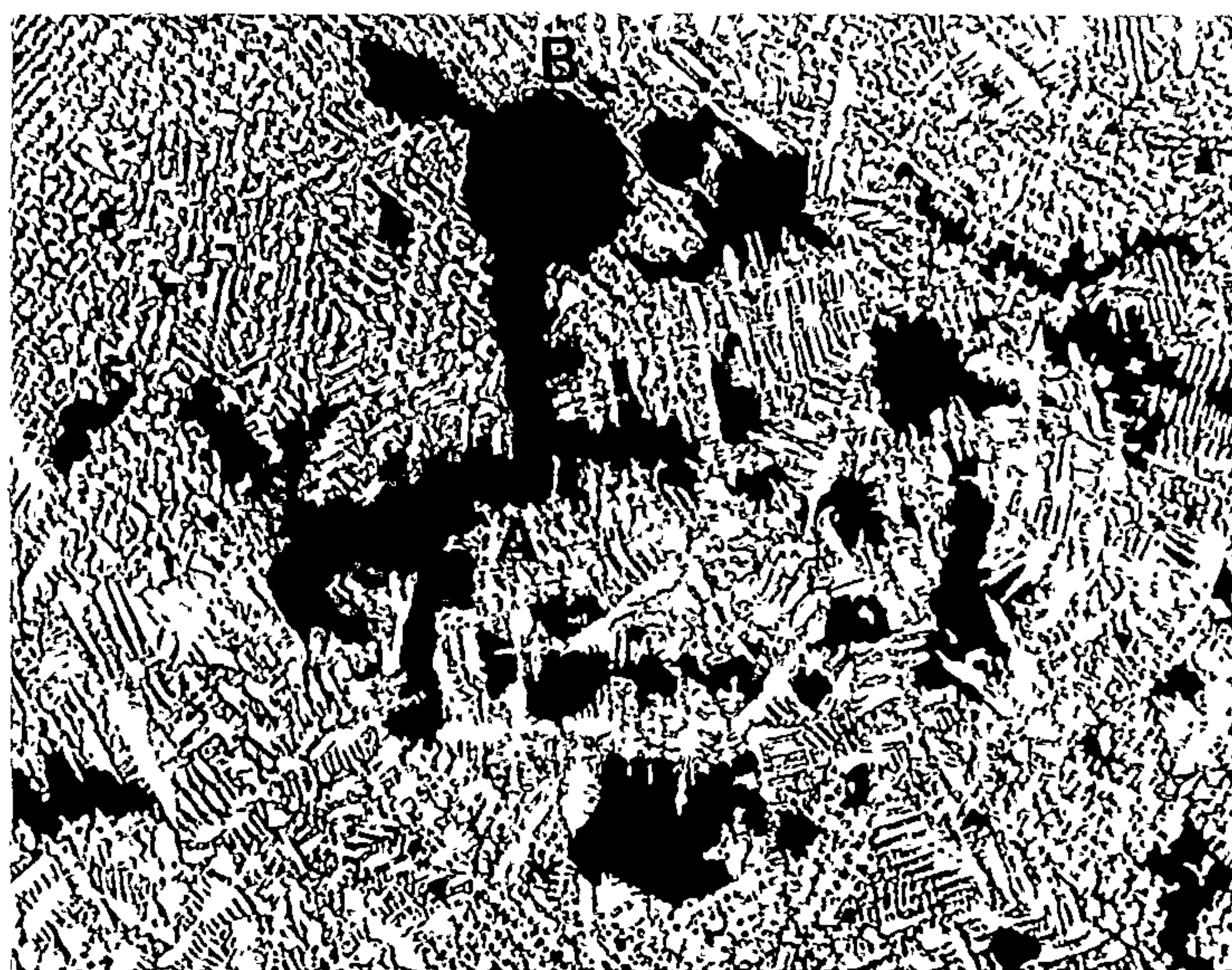




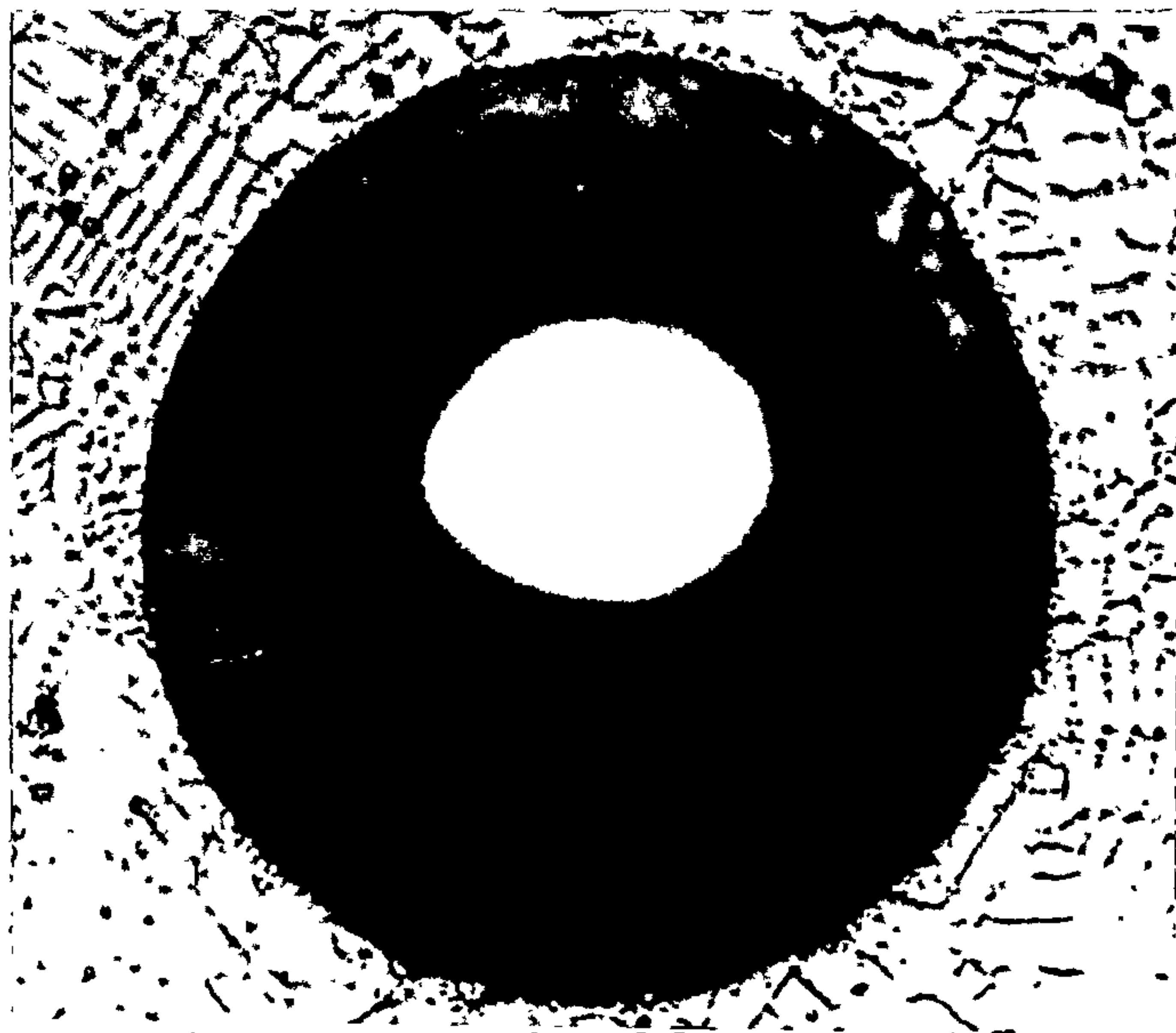
Figure 46. Type Bⁱ gold alloy(x 650).

Legend.

A. Dendritic shrinkage.

Figure 47. Type Bⁱ gold alloy (x 650).

Gaseous porosity - note sharp, well-defined, "punched out" appearance.



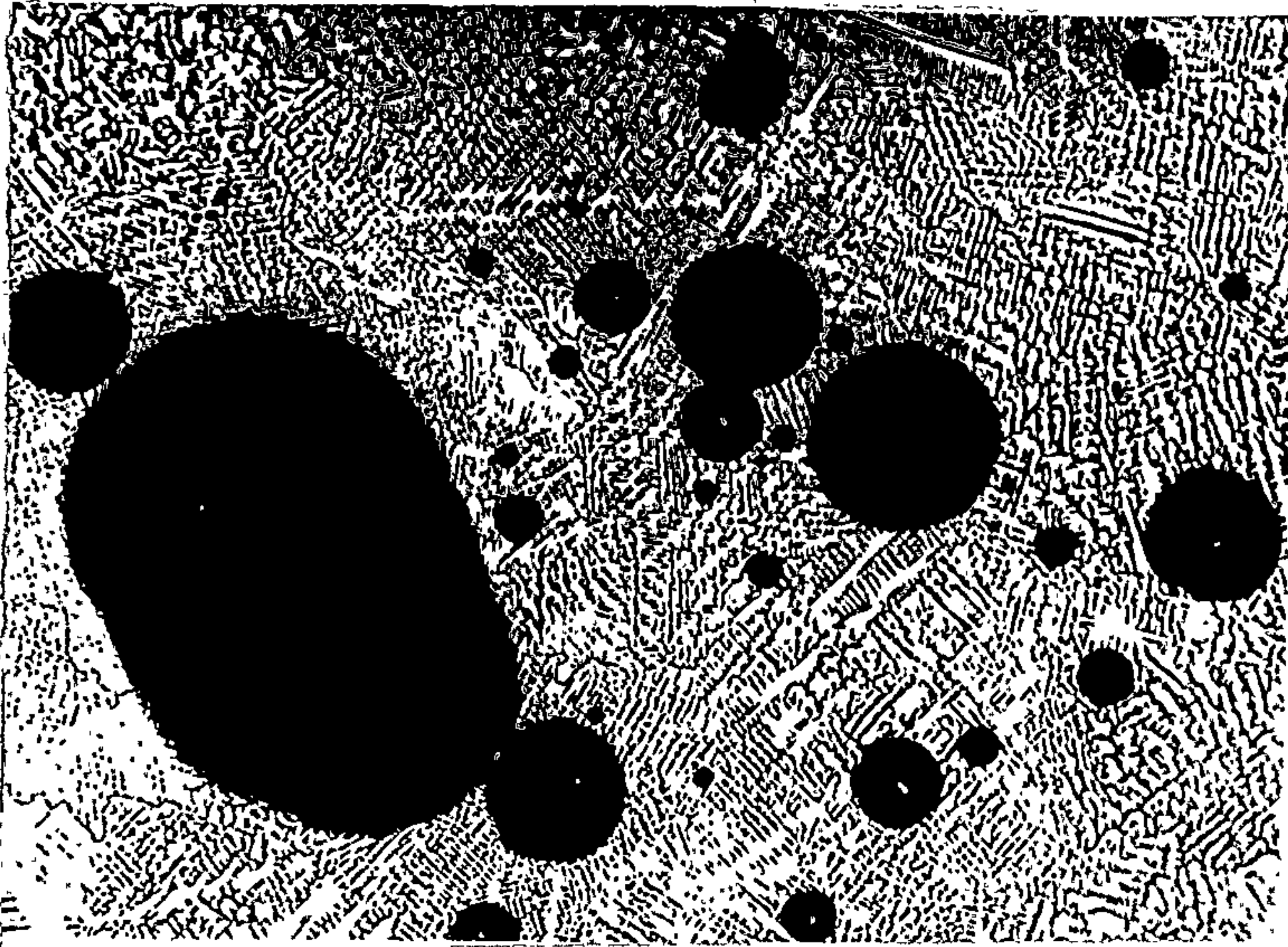
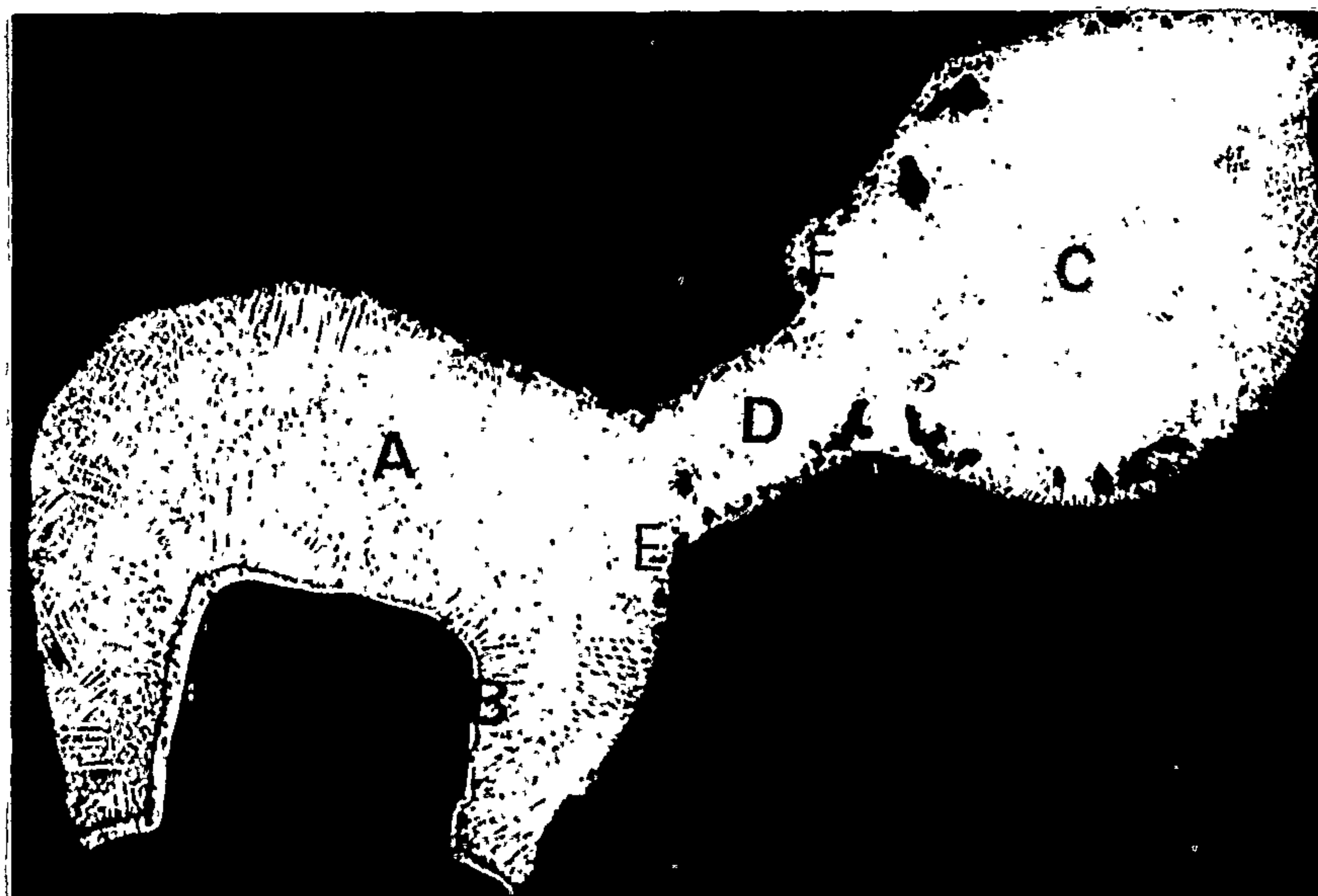


Figure 48. Type Bⁱ gold alloy showing gaseous porosity (x 350).

Figure 49. Subsurface porosity distribution of specimen seen in figure 29(x 6).

Legend.

- A. Cast gold
- B. Electroformed gold matrix
- C. Reservoir
- D. Sprue
- E. Subsurface porosity.



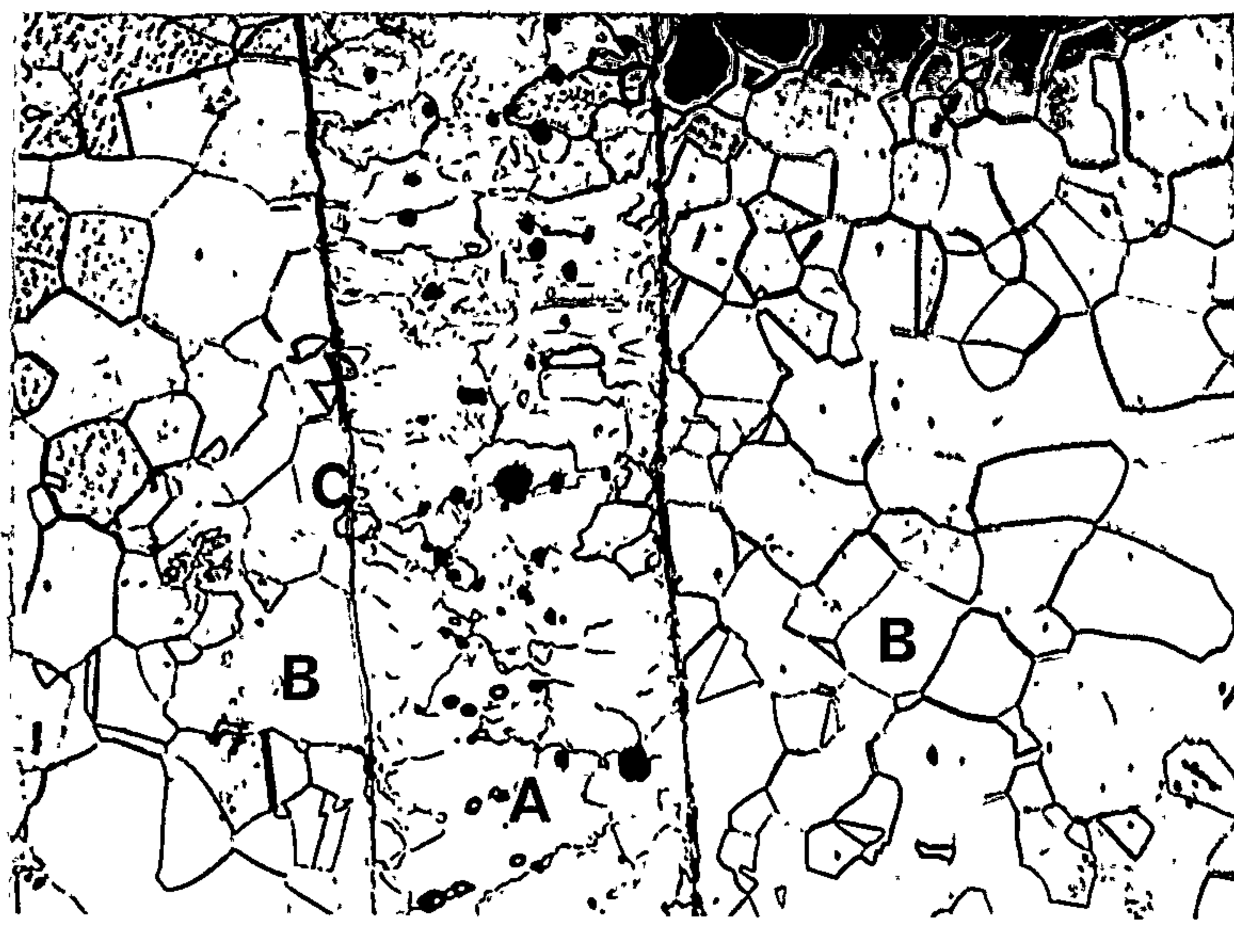


Figure 50.

Gold soldered, wrought platinised gold wire(x 200).

Quenched immediately after flow of the solder.

Legend.

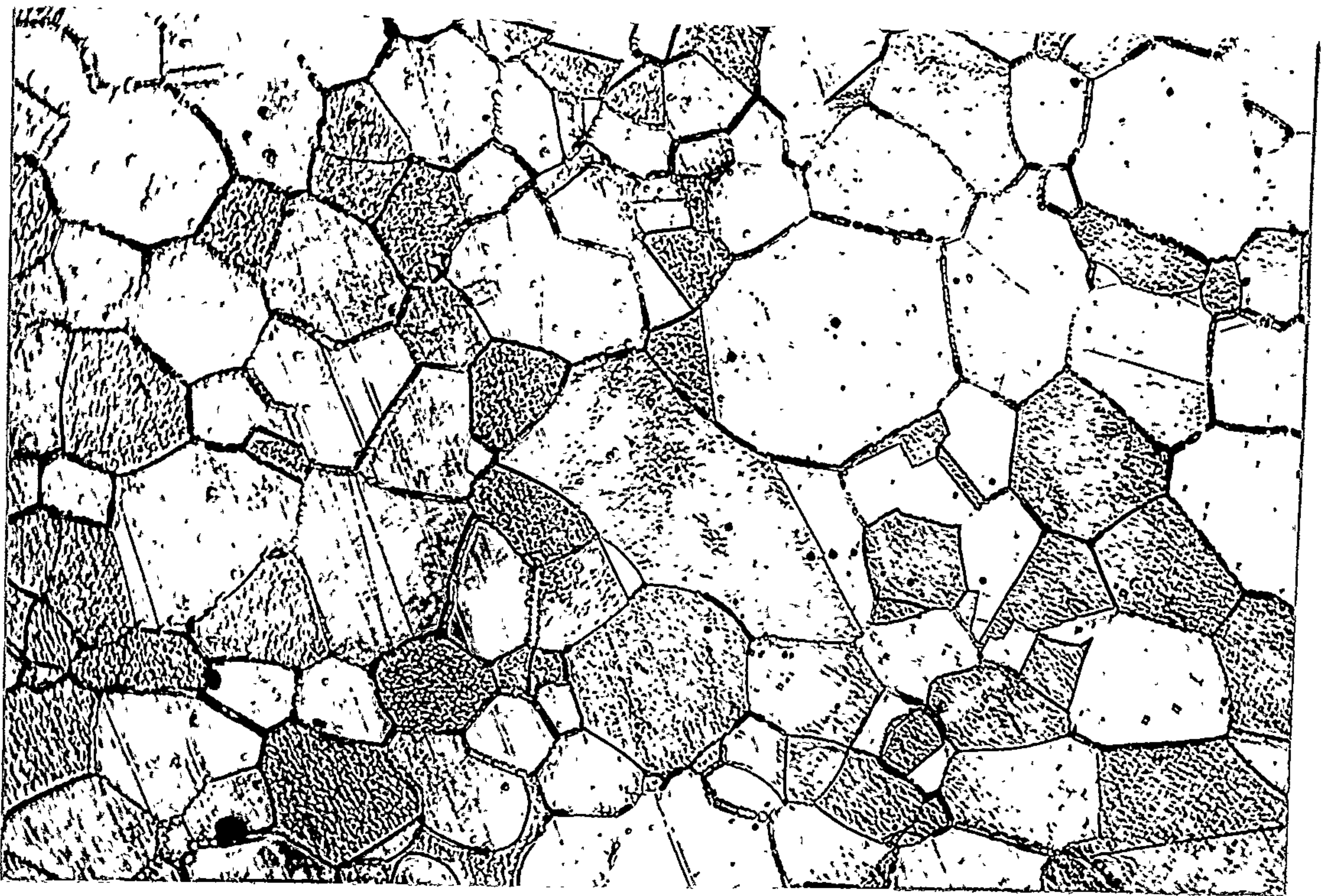
A. Gold solder

B. Gold wire

C. Epitaxy.

Figure 51A.

Wrought platinised gold wire (x 400) prior to soldering procedure.



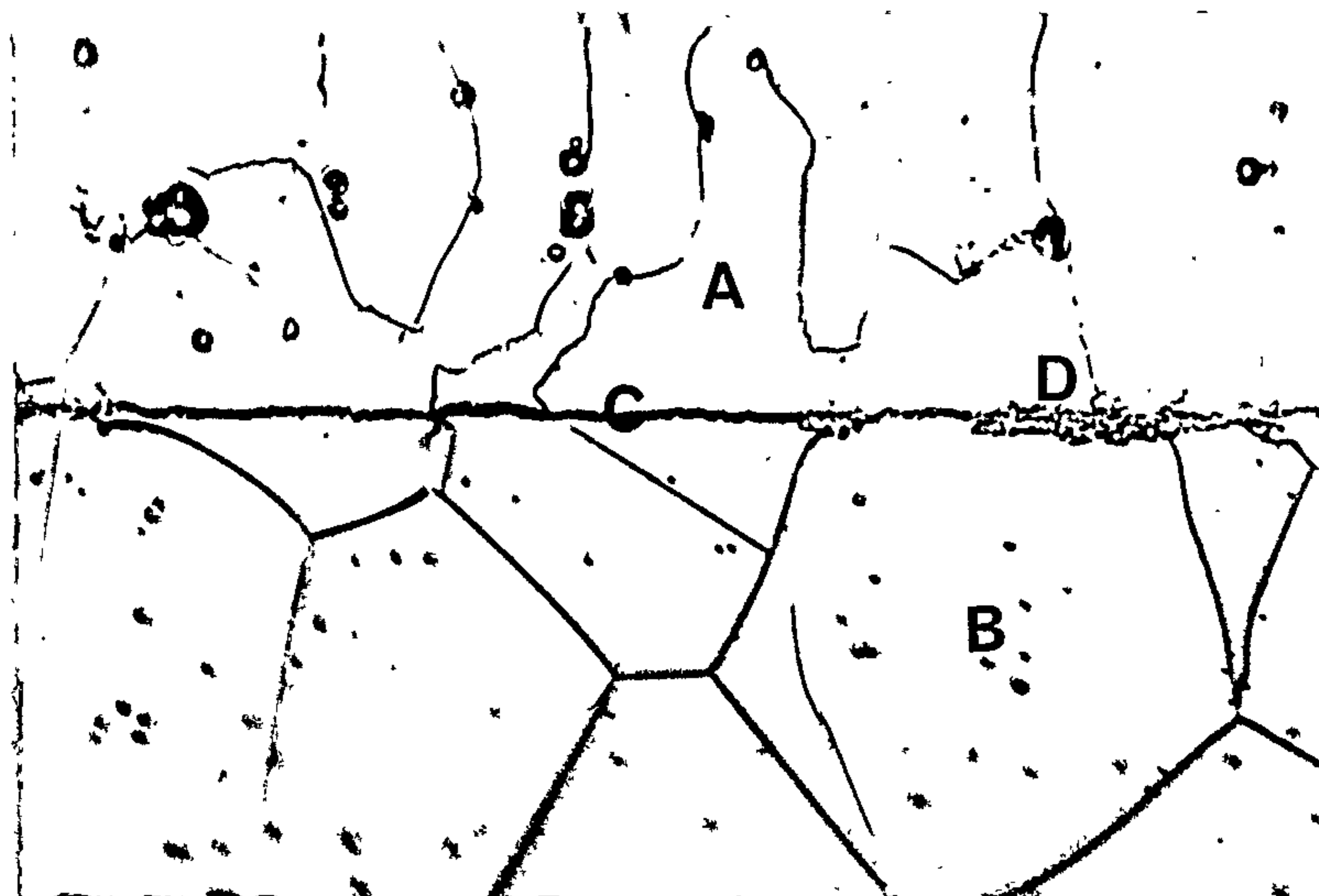


Figure 51.

Gold solder; wrought platinised gold wire (x 400).

Quenched immediately after flow of the solder.

Legend.

- A. Gold solder
- B. Gold wire
- C. Epitaxy
- D. Inclusion at interface.



Figure 52.

Gold solder, wrought platinised gold wire(x 400).

Quenched immediately after flow of the solder.

Legend.

A. Gold solder

B. Gold wire

C. Epitaxy

D. Inclusion at interface.

Figure 53.

Gold solder, wrought platinised gold wire(x 400).

Quenched immediately after flow of the solder.

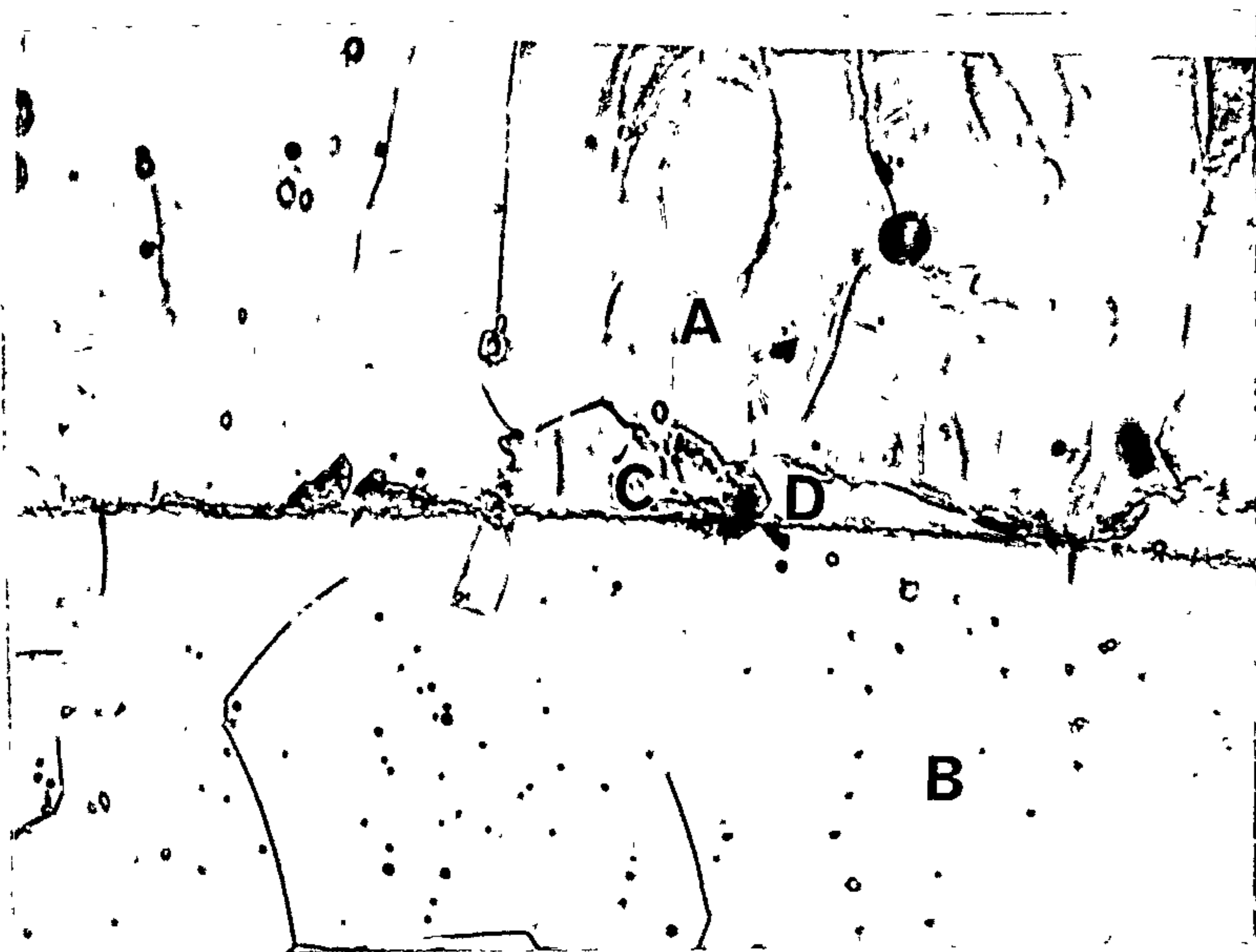
Legend.

A. Gold solder

B. Gold wire

C. Epitaxy

D. Inclusion at interface.



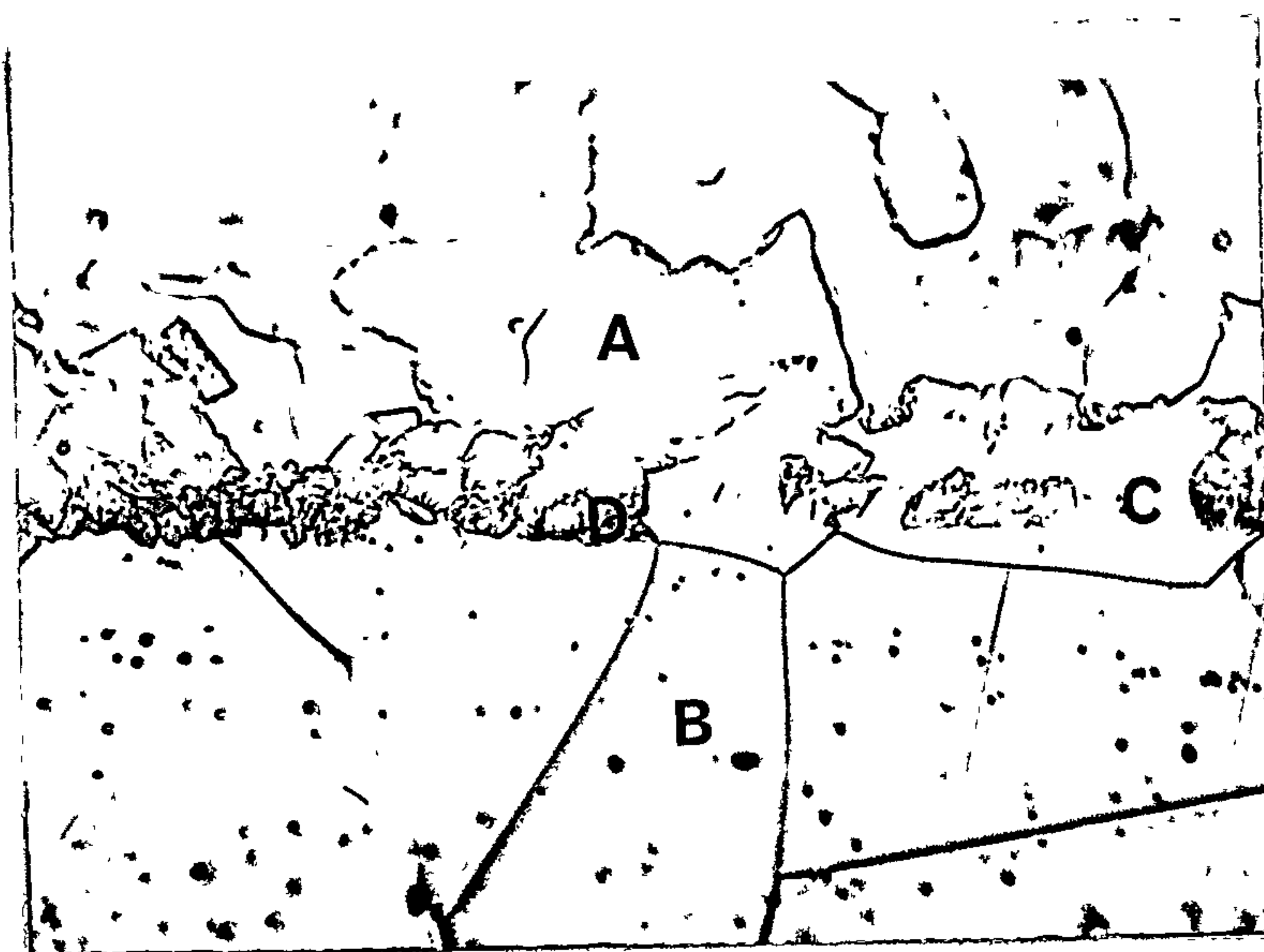


Figure 54.

Gold solder, wrought platinised gold wire (x 400).

Quenched immediately after flow of the solder.

Legend.

- A. Gold solder
- B. Gold wire
- C. Epitaxy
- D. Inclusion at interface.

Figure 55.

Gold solder, wrought platinised gold wire (x 400).

Specimen retained in the flame for five seconds after flow of the solder and bench cooled.

Legend.

A. Gold solder

B. Gold wire

C. Widening of grain boundaries.





Figure 56.

Gold solder, wrought platinised gold wire(x 400).

Specimen retained in the flame for five seconds after flow of the solder, and bench cooled.

Legend.

A. Gold solder

B. Gold wire

C. Intergranular penetration by the gold solder.

Table 1 shows the casting conditions employed for the ten doubled sprued inlays(Figures 11-14). Figures 11, 12 and 15 show a type Bⁱ gold alloy(A) cast against the electroformed gold matrix(B). A flux was used on the electroformed gold interface prior to waxing up in figures 11 and 12. The figures are of the same specimen. Figure 11 was etched in aqua regia and figure 12 was etched electrolytically.

No flux was added to the matrix surface of figure 15.

Figures 13, 14, 16 and 17 show a type C alloy(A) cast against the electroformed gold matrix(B).

The interface of the specimens used in figures 13 and 14 was fluxed. No flux was used for the specimens shown in figures 16 and 17.

Figures 13 and 16 show specimens that were etched in aqua regia, and in figures 14 and 17 the specimens were etched electrolytically.

Figure 18 shows the type C specimen that was cast and then replaced in the furnace for 24 hours at 700^oC.

Figure 19(x 400) shows the matrix area of the same specimen.

Table 11 shows the casting conditions employed for the twenty single sprued inlays that were vacuum invested and cast on a Jelenko Thermotrol casting machine(figures 20-35).

The specimens in figures 32-35 were etched in aqua regia and the remainder were etched electrolytically.

Figures 36 and 37(x 400) show the gold interface(C) between the

gold alloy (A) and the electroformed gold matrix (B) of figure 25.

Figure 38 (x 400) shows the interface of figure 27 and figure 39 (x 400) is the interface of figure 31.

Table 111 shows the results of microhardness testing (Vickers) and figure 40 (x 350) is a photomicrograph of a specimen tested for both Vickers and Knoop hardness factors.

The gold alloy as presented by the manufacturers are shown in figures 41-44. Figure 41 is a rolled, type C alloy and figures 42 and 43 show granulated gold, type B⁽ⁱ⁾. Figure 44 is a higher magnification (x 200) taken from figure 43.

Figures 45 (x 350) and 46 (x 650) are higher magnifications of dendritic shrinkage and figures 47 (x 650) and 48 (x 350) are higher magnifications of gaseous porosity.

Figure 49 (x 6) shows the distribution of subsurface porosity in the reservoir, sprue and inlay of figure 29.

Part 1.b.

The gold solder - gold alloy interface.

Figures 50 (x 200) and 51 - 56 (x 400) show the interface between gold solder (A) and wrought platinised gold wire (B). The figures 50 - 54 show soldering that was quenched immediately after flow had occurred and figures 55 and 56 show the results of retaining the specimens in the flame for an additional five seconds after flow has occurred, and bench cooling.

CONSIDERATION OF RESULTS.

PART 1. PRECIOUS METAL ALLOYS.

CONSIDERATION OF RESULTS.

Part 1.a.

Electroformed gold - gold alloy interface.

Mould and casting temperatures.

The specimens cast at a mould temperature of 550°C and a casting temperature of 20°C above the alloy's liquidus (figures 20 and 22) showed large areas of porosity (C) between the cast gold alloy (A) and the matrix (B). The union between the two metals was unsatisfactory due to incomplete venting of the mould and freezing of the alloy before all the air had been expelled. These are typical examples of back pressure porosity. A further increase of 30°C in the casting temperature considerably reduced the amount of back pressure porosity (figures 21 and 23). Figure 23, however, shows an area (D) above the isthmus of the casting, where alloy has solidified independently from the mass of the casting. The low mould temperature and the thermal conductivity of the gold matrix would have contributed to freezing in this defined area.

The specimens cast at a mould temperature of 650°C show a similar relationship - those cast at 20°C above the alloy's liquidus (figures 24 and 26) have greater evidence of back pressure porosity than those cast at 50°C above the liquidus range (figures 25 and 27). Figure 25 shows an area where the matrix has stripped away from the gold alloy (D) and also an area of dendritic shrinkage (C) at the base of the sprue. Figure 27

shows little evidence of porosity and the cast alloy and the gold matrix are in close contact.

The specimens cast at a mould temperature of 750°C at 20°C and 50°C above the liquidus (figures 28-31) show good conformation to the matrix with only small areas of back pressure porosity that would not affect the intended function and life of the inlay.

Figure 29 shows a specimen with subsurface porosity and this will be discussed later.

Figures 32 and 33 are B⁽ⁱⁱ⁾ gold specimens cast at the upper point of the alloy's melting range. Both figures show an increased amount of porosity due to gas inclusion.

Figures 34 and 35 are D gold specimens cast at the upper point of the alloy's melting range. In both these figures back pressure porosity is minimal.

The specimens cast at 700°C were flame heated so the casting temperature was uncontrolled and above the melting range of the alloy.

Figures 15, 16, 17 and 18 show limited areas of porosity and the cast gold alloy conforms to the shape of the embedded matrix.

In figures 11, 12, 13 and 14 flux was used on the matrix of the specimens. Figures 11 and 12 are mirror images of the same specimen. Marked porosity has occurred at the interface between the metals. Figures 13 and 14 are also mirror images of the same specimen, and show a similar porosity

distribution to figures 11 and 12. In figure 14 it will be observed that the matrix has perforated at E and the cast gold alloy has flowed under the matrix at F, during the casting procedure.

The porosity in these specimens is a gaseous porosity due to the release of volatile constituents when the flux and molten alloy come into contact.

These results, and the preliminary results published earlier¹³, follow those of Gibson et al.¹⁷ who cast a Type C alloy to various embedded gold alloy attachments. In their study, non-oxidising golds gave the best results and a higher mould temperature(705°C) gave better unions than lower mould temperatures. Further, it was more difficult to obtain a good union with thick metals(0.012in.-0.305mm.) than with thin metals(0.003in i.e.0.076mm.). Their test specimens were wedge-shaped and would not have provided the same conditions of metal turbulence and venting that occur when casting a more complex shaped dental restoration. Despite the thickness necessary for the electroformed gold matrix(0.008in. to 0.01in i.e. 0.2mm. to 0.25mm.) as used in this work, and despite the comparative complexity of shape, the type of union and control of porosity are comparable in both studies.

Gibson et al. used a similar investment* and casting technique, but in addition compared another investment, Hygrotrol^φ, which gave

* Kerr Cristobalite Investment.

φ Hygrotrol - Whip-Mix.

better results. This information could be applied with advantage when casting to an embedded electroformed gold matrix.

The apparent line of demarcation between the electroformed gold matrix and the cast gold alloy on some of these specimens is due to differential etching. A difference of levels between the two metals at the interface would also give a corresponding lighting effect. However particular care was taken in the preparation of these specimens to maintain a level interface and all unsatisfactory specimens were refaced.

Type of union at the interface.

For over thirty years the "Bek Process"⁹⁸ of diffusing electro-deposited gold into base metals such as nickel, copper, silver and nickel-silver alloys has been used in the jewellery trade. This technique has replaced the use of gold filled stock and rolled gold. (Gold filled stock is a sheet of basis metal such as nickel-silver with an alloyed gold "sweated" on to two surfaces. Rolled gold is similar except that only one surface is covered with gold).

Electroplated gold will diffuse into some alloys (zinc die cast and white metal) at room temperature. With less active metals heat treatment is necessary, but diffusion and alloying take place below the melting point of gold. Silver will diffuse into gold at about 480°C. An example of this is also seen in the electroformed gold matrix inlay technique when the thin adherent layer of silver metallising paint diffuses into the matrix during the casting procedure.

Kushner⁹⁸ states that the conditions for diffusion are:-

- "1. The metals must be in intimate contact.
2. The surface must be clean.
3. The metals must form a solid solution.
4. The melting point of the two metals must not be too high or too far apart."

These conditions, although applied to diffusion between an

electrodeposited metal and a basis metal, should also remain valid when intimate metal contact occurs in soldering or casting procedures. When casting to an embedded matrix it is necessary for a clean surface to exist on the matrix to ensure that intimate contact is possible between the two metals.

Figure 40 shows a Type C gold alloy (A) cast against electroformed gold (B). Knoop and Vickers hardness tests were carried out using 100g. load. Measurements were taken extending from the electroformed gold, through the interface, to the alloy. In each instance the indentation was placed as close as possible to the interface of the metals (C). Vickers hardness values ranged from 33 in the annealed electroformed gold to an average value of 145 in the gold alloy - the interface area had a value of 60. Knoop hardness numbers similarly ranged from 33 in the annealed electroformed gold to an average of 126 in the gold alloy - the interface area had a value of 55. This variation of hardness also occurred in the specimens examined and listed in Table III.

Table III shows the relationship between a number of indenting loads used on the Leitz Durimet. The statistical data presented shows that the 15 gm. indenting load was too selective where as comparable results were obtained with both 50 and 100 gm. loadings which gave more averaging impressions.

The hardness impressions are indicative, but not conclusive proof, of diffusion.⁹⁹ Hardness numbers cannot be accepted as proof of diffusion or interalloying as the indentation is greater than the area being

investigated and the indenting head involves areas on either side of the metal union. However, as a hardness indentation with a reasonably symmetrical form is produced at the metal to metal interface and the gold matrix has not stripped away from the cast gold, some form of attachment between the metals is indicated.

Figures 36, 37, 38 and 39 show the interface between the two metals at higher magnification (x 400).

Figure 36 shows a type Bⁱ gold alloy (A) cast against the gold matrix (B) and the interface (C) between the two metals. Oblique lighting was used. There may be some diffusion in limited areas indicating the continuation of structure from one area to another but the general appearance is similar to that of a gold soldered joint where wetting has occurred.

Figure 37 is a similar alloy (type Bⁱ). Pinhole porosity is present in the cast alloy (D) but the union between the two metals is sufficiently strong to produce a satisfactory inlay. The line of demarcation (C) between the alloy (A) and the electroformed gold (B) has become indefinite and diffusion appears to have occurred between some grains in the lower part of the photomicrograph (E).

Figures 38 and 39 show a Type C gold alloy (A) as cast against the electroformed gold matrix (B). A bond is evident between the metals (C) and wetting with diffusion has occurred at the interface. Figure 38 (D) shows the presence of some form of inclusion.

In figure 18 a Type C gold alloy was cast against the matrix and

the invested inlay replaced in the electric furnace for 24 hours at 700°C . Complete diffusion has occurred and the gold matrix cannot be identified. Figure 19 shows the interface area of this specimen at a higher magnification (x 400). Only a vague trace of the gold matrix (B) remains and the interface (C) has completely disappeared. Inter alloying has occurred and the diffusion has involved both sides of the interface - from the matrix into the alloy and from the alloy into the matrix. Both metals (A and B) have lost their characteristic structure.

Braun has also reported reaction-rate studies between gold and solders of lead-tin-indium and lead-tin-indium-zinc alloys. After 1 hour at 160°C gold wire measuring 0.81mm. in diameter was completely consumed by the solder.¹⁰⁰

Bonding by diffusion does occur between electroformed gold and cast gold inlays. The heat saturation time of five minutes after casting produced an adequate bond between the metals and prolonged heat treatment caused inter alloying.

Inclusions at the interface.

There is no microscopical evidence of residual by-products of electrodeposition or residual carbon from the wax elimination procedure remaining between the cast gold alloy and the electroformed gold. This is supported by the close union, with evidence of diffusion, that occurs at the interface and the lack of surface contaminants, or inclusions, that make the use of a flux unnecessary when casting to the matrix.

Porosity.

Porosity is a feature of all gold castings. Skinner and Phillips¹⁰¹ broadly classified porosities into two groups - external and internal.

The external porosities are usually evident on macroscopic examination and include back pressure porosity, incomplete burnout, and porosity due to incomplete casting pressure and foreign particles.

The internal porosities are caused by the method of solidification of the metal and by occlusion of gas whilst the metal is molten.

The most common cause of porosity is back pressure porosity¹⁰¹. Gases from both the investment and the atmosphere are present in the mould and during casting, the molten metal must force this gas out through the investment. If the gases cannot be dissipated they form a back pressure which, if sufficiently strong, will force the gases back into

the molten metal to create voids on solidification.^{102,103,104}

Skinner and Phillips state¹⁰¹ that with "the modern dense investments, the increase in mold density effected by vacuum investing and the tendency for the mold to clog with residual carbon when the low heat technic is used, all increase the difficulty in venting the gases from the mold during casting."

In addition to these factors the presence of the gold electroformed matrix in the mould offers a further resistance to venting.

According to some authorities vacuum investing does not increase back pressure porosity,^{104,105} as the large air bubbles in the investment are broken down into multiple small voids to provide even higher permeability. Shell and Dootz¹⁰⁵ found that these small voids provided less restriction to the passage of air and that a thickness of 0.25 inch of investment should allow 0.25c.c. of gas to escape in less than one second.

Kelly¹⁰⁶ found that molten metal when cast starts to solidify in 3 to 4 seconds and is solid in 5 to 6 seconds.

To control back pressure porosity adequate time must be allowed for venting to occur whilst the metal is in the molten state, and an adequate casting pressure must be maintained during this period of time.^{102,103,105}

Similarly insufficient casting metal will lead to a back pressure porosity due to reduced casting pressure.¹⁰²

Venting by using an additional vent hole has been advocated^{102,107} but the use of a sprue long enough to place the outer end of the casting

within 6mm. of the open end of the ring is considered adequate.^{102,104,108} Strickland and Sturdevant¹⁰² recommend a high temperature casting technique to eliminate back pressure porosity. It is generally agreed that higher mould temperatures reduce porosity in castings.^{101,104,106,109} Two examples of back pressure porosity are seen in figures 20 and 22. They occurred when casting at a low mould temperature (550°C) and a low casting temperature (20°C above the liquidus of the alloy). At higher mould and casting temperatures the embedded matrix did not restrict venting to the extent of creating a back pressure porosity - sufficient time existed for the alloy to pass from the liquid to the solid phase whilst under adequate casting pressure.

The other causes of external porosity i.e. incomplete burnout, porosity due to incomplete casting pressure and foreign particles, occur infrequently and are usually due to an accidental deviation from accepted casting procedure.

Ryge, Kozak, and Fairhurst¹¹⁰ classify the internal porosities found in gold castings into two groups - those formed by cooling and solidification and those caused by gas.

Before discussing dendritic and gaseous porosities it might be opportune to examine these types of porosity and determine their differences.

Photomicrographs 45 and 46 show areas of dendritic shrinkage, with a possible gaseous inclusion (B) in figure 45.

This type of porosity is characterised by the sharp, irregular nodules or spines of the dendrites that give a ragged, labyrinthine appearance to the internal surface of the voids so formed.

When molten a gold alloy occupies more space than after it solidifies and a value of 1.25 ± 0.1 percent has been calculated for this shrinkage which occurs during a casting procedure.¹¹¹

Crawford¹¹² describes how the alloy commences to freeze at the upper end of its melting range and dendrites form and grow until they collide into adjacent dendrites. After the growth of the dendrite is arrested the remaining molten metal fills in around the dendrite. As freezing continues, with an accompanying contraction, there is insufficient metal to completely fill in around the originally formed framework. This results in voids around the spines of the dendrites in that portion of the casting that is last to freeze.

Gaseous porosity as seen in figures 47 and 48 has quite well defined margins with a sharp clean punched out appearance. The surface of the void appears as if a complete round grain, with its grain boundary, has been removed.

Porosities caused by solidification and shrinkage.

a. Localised shrinkage porosity.

This type of porosity is caused by a lack of molten metal during solidification of the casting. When an inlay is cast the metal is forced from the molten button through the sprue into the mould. Freezing occurs most rapidly in the thin portion of the mould where a small amount of

alloy is in contact with a large area of the investment.¹⁰⁹ The inlay should freeze before the sprue and the button (or reservoir if used) so that metal is available to compensate for the shrinkage that occurs during solidification.^{110,111,112}

The use of a long or thin sprue leads to an increase in porosity.^{103, 106,109,110,112,113,114} Voids formed in the molten gold during solidification cannot be replenished if the gold is frozen within the sprue.

Localised shrinkage porosity usually occurs at the point of sprue attachment to the inlay, and in the case of M.O.D. type inlays, at the isthmus joining the two proximal surfaces.¹¹⁵

The sprue diameters, length and placement used in the production of the casting specimens did not cause localised shrinkage porosity except in figure 25 where a small area of dendritic shrinkage occurred.

b. Microporosity.

This is characterised by small irregular voids throughout the entire casting caused by rapid solidification of the cast alloy due to a low mould and, or, casting temperature.

Thus Ryge et al.¹¹⁰ consider microporosity as a generalised form of localised shrinkage porosity. Instead of the sprue freezing to form a localised dendritic shrinkage near the sprue attachment, solidification occurs so fast that shrinkage develops throughout the casting. It was observed "throughout castings made in molds of 500°F (260°C) or room temperature and with an alloy temperature within or slightly above the

Reproductions. (Ryge, Kozak and Fairhurst.)¹¹²

Reproduction 1.



Fig. 1 • Spherical gold castings. a, localized shrinkage porosity; b, microporosity; c, pinhole porosity and d, subsurface porosity

Reproduction 2.

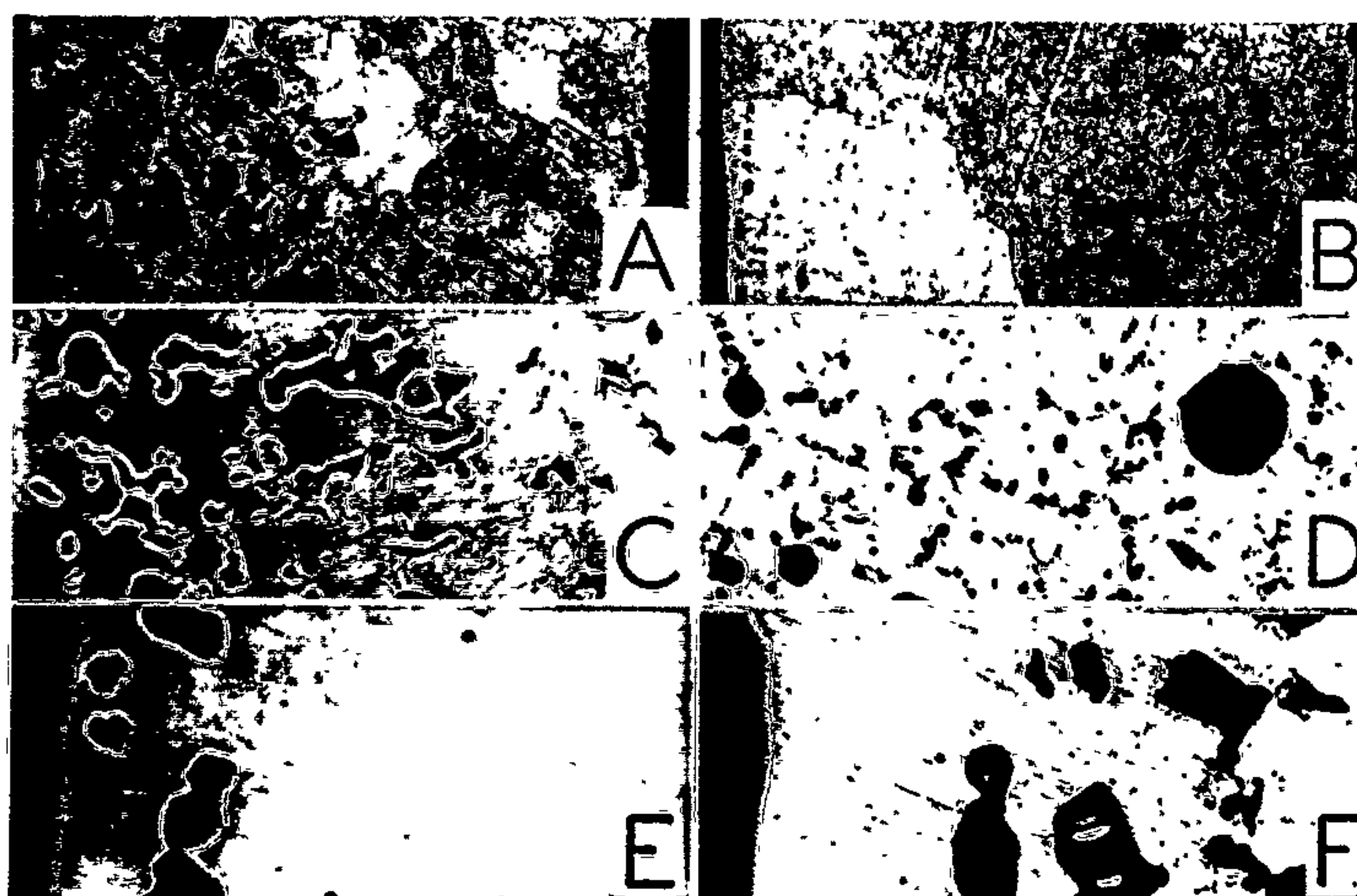


Fig. 2 • A: Grain size in casting made with 3 mm. (short), 10 gauge (thick) sprue. B: Grain size in casting made with 9 mm. (long), 10 gauge (thick) sprue. C: Localized shrinkage porosity (see also Figure 5,A). D: Microporosity and gas inclusions (see also Figure 5,D). E: Subsurface porosity (see also Figure 6,A). F: Subsurface porosity (see also Figure 6,B)

Reproduction 3.

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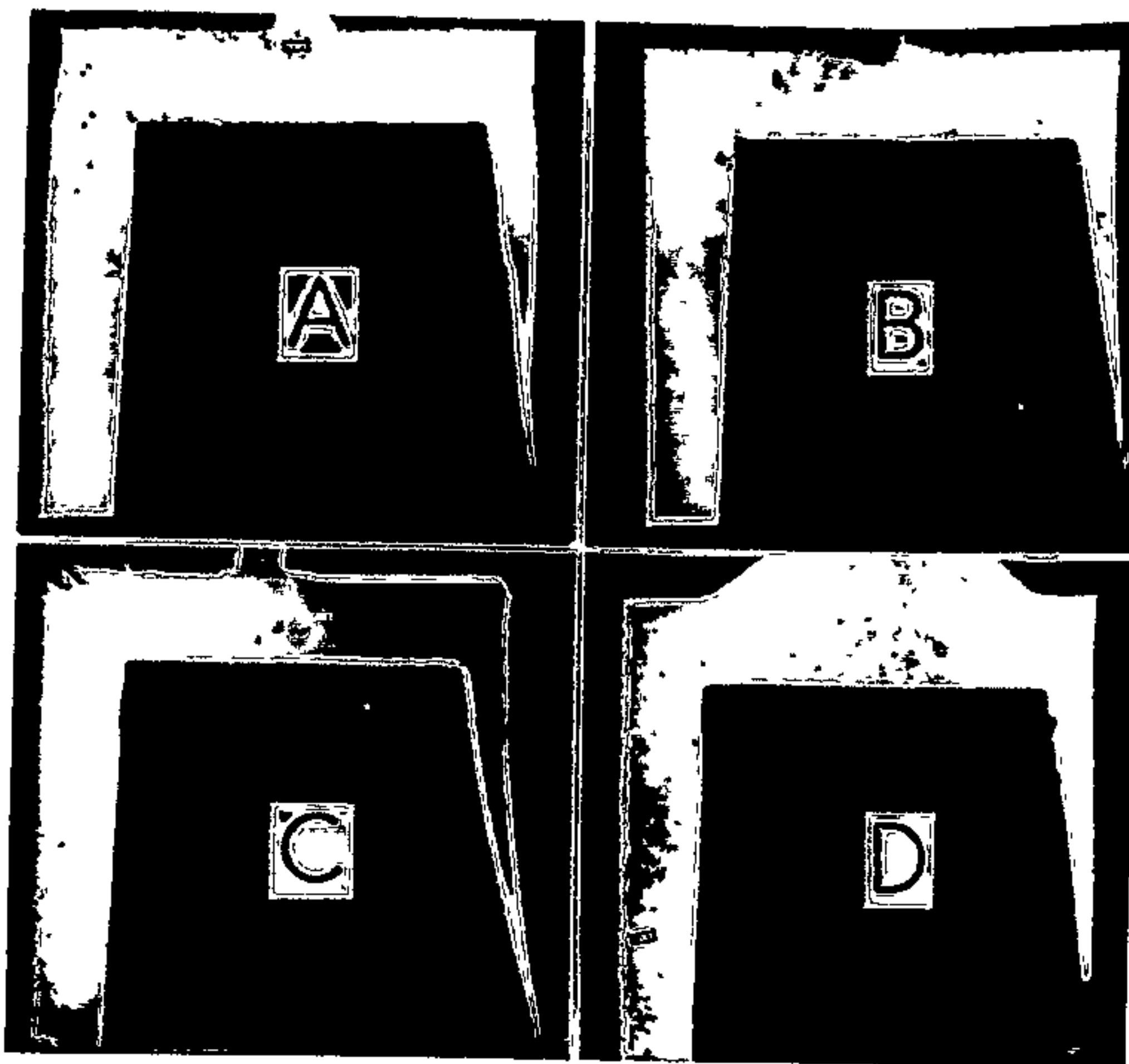


Fig. 5 - A: Shrinkage porosity in casting made with 3 mm. (short), 18 gauge (thin) sprue. Area outlined is shown in higher magnification in Figure 2,C. B: Localized shrinkage porosity in casting made with 9 mm. (long), 18 gauge (thin) sprue. C: Pinhole porosity throughout casting made with alloy heated above 2,000°F. D: Microporosity and gas inclusions (large, circular pores) in casting made with alloy heated slightly above its melting range. Area outlined is shown in higher magnification in Figure 2,D

Reproduction 4.

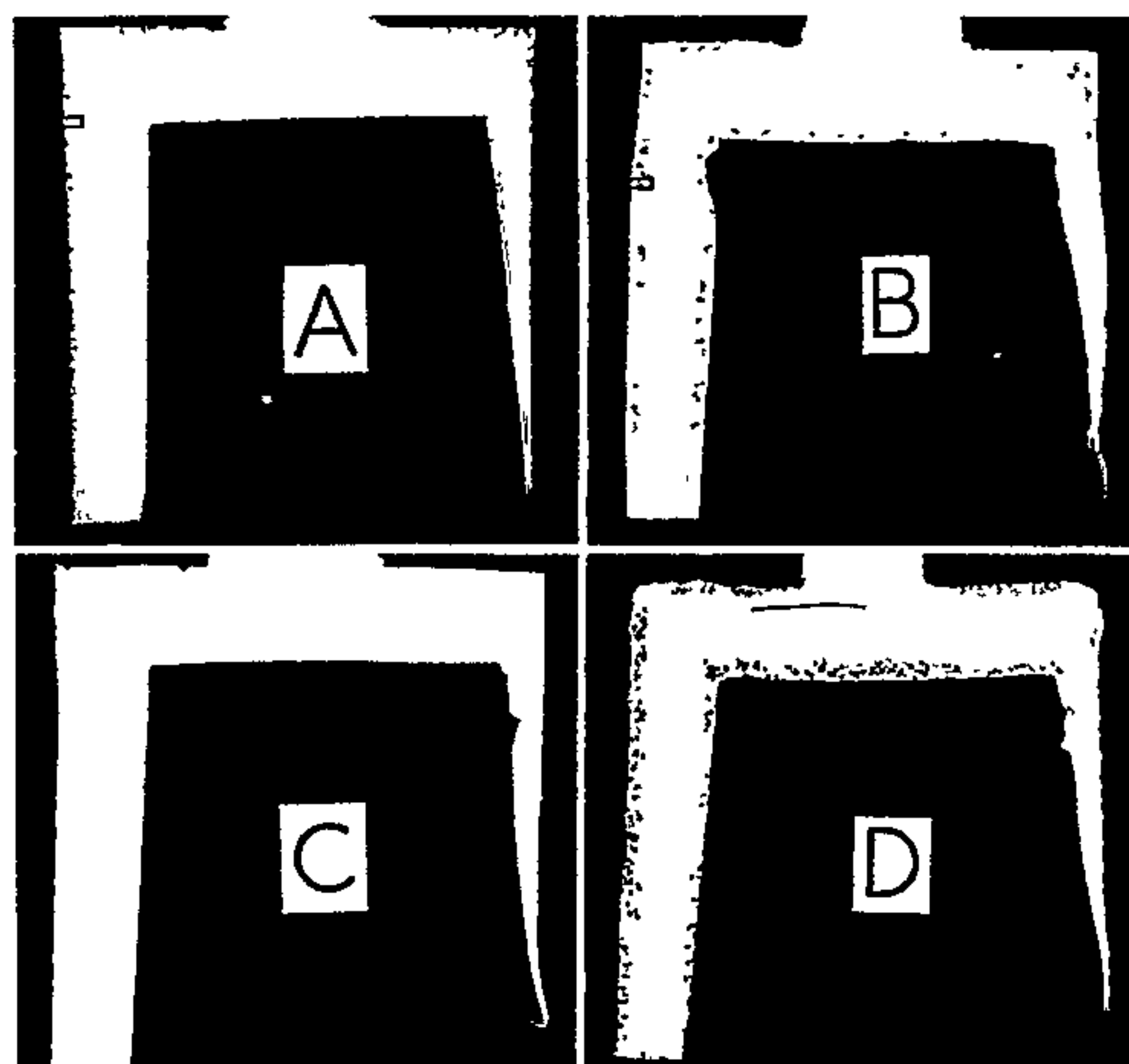


Fig. 6 - A: Subsurface porosity in casting made with 3 mm. (short), 10 gauge (thick) sprue. Area outlined is shown in higher magnification in Figure 2,E. B: Increase in amount of subsurface porosity in casting identical to that in A except for increase in alloy temperature. Area outlined is shown in higher magnification in Figure 2,F. C: Sound casting made with 9 mm. (long), 10 gauge (thick) sprue. D: Increase in amount of subsurface porosity in casting identical to that in A except for increase in mold temperature

Reproduction 5.

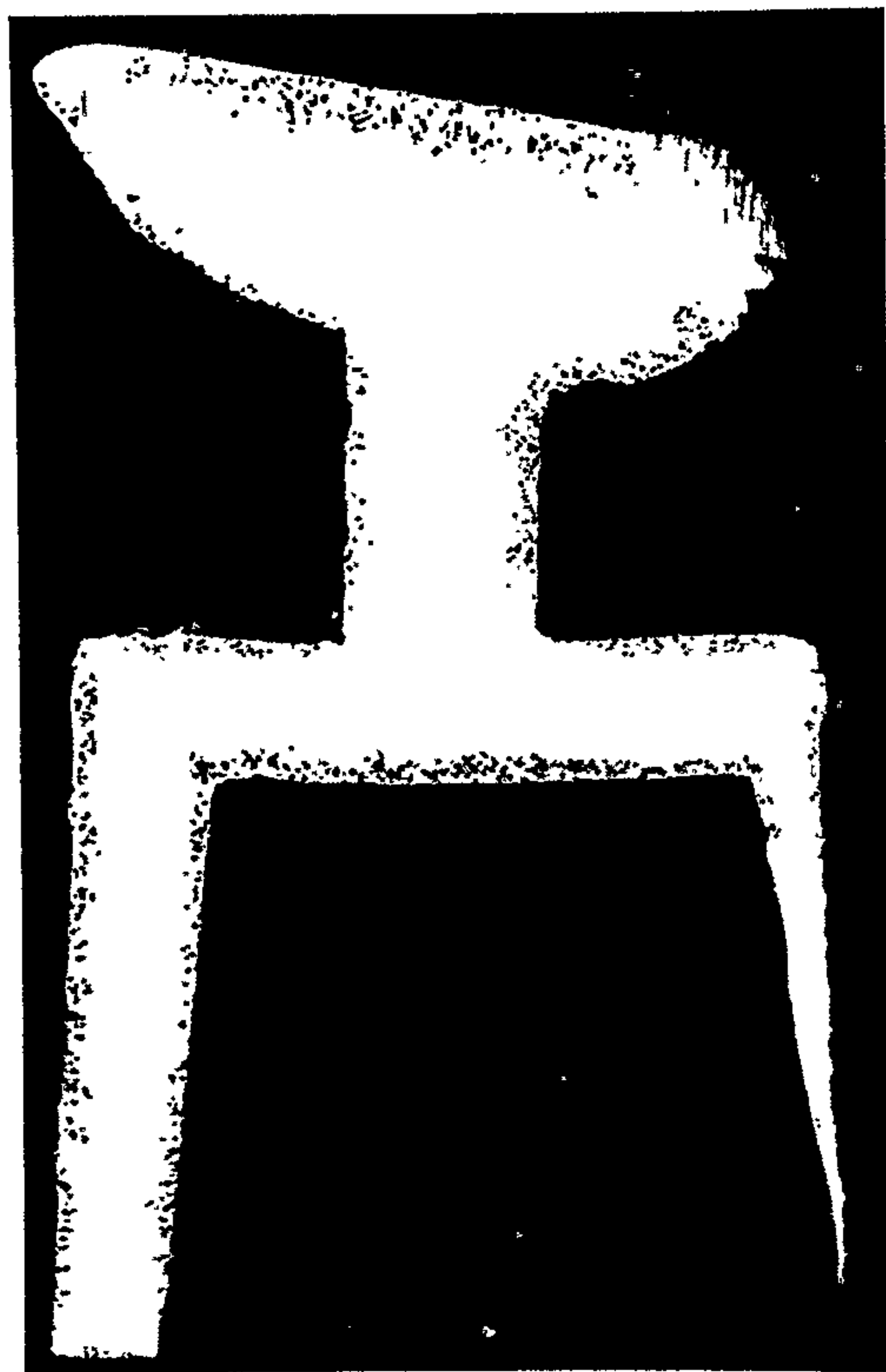


Fig. 7 • Same specimen as in Figure 6,D, showing subsurface porosity near all surfaces of casting, sprue and button

melting range." No relationship existed between the sprue size and microporosity.

This type of porosity will be discussed further in relationship to pinhole porosity.

c. Subsurface porosity.

According to Ryge et al.¹¹⁰ subsurface porosity occurs near the border of the casting and is thought to be related to the rate of solidification of the casting (reproductions 1, 2, 4, and 5). If the molten metal is fed into the mould rapidly at a high temperature the skin of the casting solidifies and the centre mass of the alloy remains liquid for an interval before it solidified. The surface skin is locked to the walls of the mould and when the central portion contracts during solidification, it leaves voids between it and the skin.

The porosity is characterised by its formation in the interstices of the dendritic structure and it is stated that if it was a type of gas porosity it would be distributed uniformly throughout the casting. Slower feeding (long or thin sprues) or lower alloy or mould temperatures eliminated subsurface porosity because rapid solidification occurred throughout the casting on contact with the mould.

The only example of subsurface porosity that occurred in all the castings investigated is shown in figure 49 (Figure 29 is the same specimen). The subsurface porosity is limited to the reservoir, sprue and upper regions of the inlay and it is a gaseous porosity although the

areas of porosity are irregular and not typically spherical an outline. The matrix area is free from subsurface porosity and the internal areas of the casting adjacent to the matrix are unaffected.

The invested gold matrix is a metallic skin. Heat is conducted from the molten alloy, which is $20-50^{\circ}\text{C}$. above the liquidus in the controlled castings, and a variable with the flame-melted castings. Wetting and solidification occur quickly and the keying effect of the 0.08 - 0.01in.thick matrix would be greater than that obtained between the solidified skin of alloy at the mould surface. These conditions should fulfil the requirements for subsurface porosity if it is of a dendritic nature.

In figure 23 an area of alloy(D) has solidified separately from the bulk of the alloy. The conditions for a subsurface porosity of a dendritic nature as described by Ryge et al. have existed in this area but it has not occurred.

In reproduction 5 subsurface porosity is present beneath the upper surface of the cast button. The skin formed on solidification would not be reinforced by the mould in this situation, yet the area of subsurface porosity is heavy and dispersed. The upper surface also shows no sign of contraction towards the dendritic shrinkage despite the assistance from the centrifugal force that is applied during the casting procedure. This would indicate a restricting internal pressure such as gaseous porosity, rather than the internal shrinkage porosity of a dendritic nature.

The porosity in reproduction 2 (E & F) as well as figure 49 does

not specifically resemble dendritic shrinkage. Ryge et al. consider that subsurface porosity is "characterised by its formation in the interstices of the dendritic structure." This statement can be related to all types of internal porosities.

The examples in reproductions 4 and 5 relating to subsurface porosity have short thick sprues and a distance of 3mm. between the pattern and the casting reservoir. Consequently the pattern would be an addition 6mm. from the base of the casting ring when compared to reproduction 4C which shows a sound casting, implying difficulty in venting the mould.

Reed-Hill states¹¹⁶ that the solubility of gases in metals is also a function of temperature and in most cases the solubility increases rapidly with the temperature. Similarly changes in the solubility of gases in metals occur at the freezing point so that when a metal freezes its ability to hold gases is strongly decreased. Gas bubbles usually form heterogeneously and in most cases the nucleation at the interface is also furthered by the build up in concentration of the gaseous solutes at both the general interface and in between the dendritic arms as a result of segregation effects. The normal shrinkage that occurs when a metal freezes causes a pressure drop in the liquid. This situation may occur when the entire outer surface of the casting freezes over leaving the liquid in the centre surrounded by a shell of solid metal. A vacuum may develop and if it does, gas-bubble and nucleation will be promoted.

This would explain the phenomenon of subsurface porosity. The irregular shaped gaseous voids are related to dendritic shrinkage but are gaseous in nature.

The metallic components of gold alloys such as silver, copper and palladium are highly susceptible to oxygen and hydrogen inclusions.^{109,112,117}

Additional gas may also be entrapped in the molten gold alloy due to the short, heavy sprue as previously discussed. The increase in subsurface porosity due to an increase in casting temperature would relate to the increased amount of gas dissolved in the molten alloy. The thin shell of solid metal surrounding the cast metal would prevent escape of the occluded gases and the effect of dendritic shrinkage between the solidified metal and the molten core would both promote and control the shape of the gaseous porosity.

Porosities formed by gas.

a. Pinhole porosity.

Gases absorbed during the melting of the alloy are expelled during solidification and dispersed throughout the casting as a pinhole porosity. This type of porosity occurred mainly when natural gas-air was used for melting the alloy at high temperature.¹¹⁰

Reproductions 1 and 2 show microporosity and pinhole porosity. Figures 11, 13 and 16, were etched in aqua regia and have a similar appearance to those of Ryge et al. Figures 12, 14 and 17 are the same specimens after electrolytic polishing and etching.

Most of the pinhole porosity in figures 11 and 13 has become obscured in the etched structures shown in figures 12 and 14. However certain areas

on these specimens (D) have assumed a dendritic type of structure.

This is more definite in specimens 16 and 17 where much of what appears to be pinhole porosity assumes a dendritic type porosity - microporosity - after electrolytic polishing and etching.

Pinhole porosity can be seen quite distinctly in figures 36, 37, 39 and 40 at the higher magnification.

From this it can be concluded that microporosity occurs at higher mould temperatures than those previously reported, its presence being masked by pinhole porosity-like voids from which it could not be differentiated by the techniques previously available. To clearly distinguish pinhole porosity from microporosity electrolytic polishing and etching techniques are required and high power microscopical examination is necessary.

b. Gas inclusions.

Absorbed or mechanically trapped gas that is carried into the mould by the alloy during the casting procedure is classified as gas inclusion. These inclusions resemble pinhole porosity except the voids are usually larger. Ryge et al.¹¹⁰ found that gas inclusions occurred when the alloy was melted with natural gas-air and was most extensive when the alloy was heated only slightly above its melting range.

A unique type of gas inclusion is shown in figures 11, 12, 13 and 14. The gaseous inclusions (C) are due to a flux used on the matrix surface which volatilised when contacted by the molten metal.

It also seems reasonable to relate gas inclusions and back pressure porosity. The casting of an alloy at temperatures close to its melting range would lead to earlier freezing of the alloy. As previously

mentioned it takes almost one second for a mould to vent under favourable conditions.¹⁰⁵ It does not seem unreasonable to expect that during this period of time some of the gas within the mould must penetrate back into the molten alloy and be trapped by the surface skin formation as the alloy begins to freeze. Back pressure porosity could belong to both the internal and external types of gaseous porosity.

The classification as presented by Ryge et al. has long been the major work in the field of internal porosities in dental castings. However a review of the incidence of microporosity and pinhole porosity and a reclassification of subsurface porosity is indicated using modern techniques of metallographic examination.

Spruing.

As previously discussed in the section on porosity it is generally agreed that the use of heavier sprues lead to a reduction of porosity in dental castings.^{102,103,106,110,112,113} Leinfelder, Fairhurst and Ryge¹⁰⁹ used a qualitative method for evaluating the amount of porosity in gold castings. They determined the specific gravity of cast inlays and concluded that the apparent specific gravity (amount of porosity) is dependent on the thickness of the sprue, the length of the sprue and the temperature of the mould. As higher mould temperatures are necessary^{102,104,106} and the length of the sprue determined by the venting requirements^{102,108,114} then the only variable remaining is that of thickness.

The results obtained with the double 14 gauge (1.7mm.) and the single 10 gauge (2.6mm.) sprues at high mould and casting temperatures in this

series of experiments have been discussed and are comparable.

Skinner and Phillips¹¹⁸ state that "the direction of the sprue former is also important. It should never point directly toward a thin or delicate part of the mold because the molten metal, as it rushes in, may abrade or fracture the investment in this area and a casting failure may result."

This problem does not apply when casting to a matrix. The matrix is opposite the sprue entrance so that the ingressing gold strikes the matrix and not the mould surface.

In all castings it is important to place the sprue at the point of greatest bulk in the pattern.¹¹⁸

The reasons given are:

- a. To reduce distortion in the wax pattern
- b. To prevent localised shrinkage porosity.

The first reason does not apply to the electroformed gold matrix inlay technique as the wax is reinforced against distortion by the gold matrix. However a second reason must be observed as the area of greatest bulk is the last to solidify and the sprue should supply additional metal during the freezing and shrinkage stage to control porosity due to dendritic shrinkage.

Investing Procedure.

It is generally acknowledged that equally good results can be obtained with hand investing as with vacuum investing.^{104,109,120}

However the latter method is probably more dependable in the

prevention of surface nodules. Air bubbles can become attached to the surface of the pattern during the investing procedure leading to surface irregularities on the casting. An example of this is seen in figure 32.

Surface imperfections on gold castings may damage frail enamel margins and, if on the cavity fitting surface, prevent proper seating of the inlay.

Fortunately this factor is not as important when using the electro-formed gold matrix inlay technique as the matrix is the tooth fitting surface. However casting discrepancies should be minimised to simplify polishing and finishing procedures.

In comparing the results of the two investing procedures used, there is no clear delineation showing which is the better except that greater standardisation is possible with the vacuum investing technique and it reduces the demands on the operator's ability.¹²¹

Melting and casting the alloy.

The melting of the alloy by using the reducing zone of a gas-air flame, or within an electric muffle did not influence the final results. Similarly there is no gross distinction between the handswung and the machine cast specimens. This also has been reported by other investigators.^{104,120}

Skinner and Phillips¹²¹ state that "any method is satisfactory if the molten alloy can be forced into the mold under sufficient pressure so that the pressure can be maintained for at least four seconds after the gold has been cast."

However, the use of the electric muffle for melting the alloy and a casting machine for introducing the molten metal into the mould eliminates variables that can occur in techniques dependent on the operator's experience.

Porosity in rolled and granulated gold alloys.

Figure 41 shows rolled Type C alloy, and figure 42 a granulated Type B⁽ⁱ⁾ alloy. The fine-grained structure of the rolled gold and the radially grained structure of the granulated gold conform to the expected appearance.

Figure 43 is also granulated Type B⁽ⁱ⁾ alloy as supplied, and the specimen shows areas of occluded gas. The incidence of porosity is reduced in rolled gold alloys as the deficiencies that occur during manufacture are collapsed and weld together. Leinfelder, Fairhurst and Ryge¹⁰⁹ report that after three meltings and three solidifications gold alloys reach and maintain a constant specific gravity i.e. a state of "porosity equilibrium".

Most of the occluded gas in specimens such as figure 43 would be liberated when the alloy was in the molten state, and the density of the gold would displace some of the remaining gas when cast. However its presence could be considered as an additional casting hazard.

Figure 44 shows the dendritic and equiaxed structure of type B⁽ⁱ⁾ gold alloy (X 200) with only limited areas of pinhole porosity. Note the typically random orientation of the grains, or crystals. Porosities cannot be excluded from casting procedures so alloys should be prepared and supplied with a minimum of such defects.

Part 1.b.

The gold solder - gold alloy interface.

The first property listed by Ryge²³ in classifying the desirable properties of gold solders is that they must be free flowing. This refers to the location of the fusion range which must be lower than that of the parts being soldered. Skinner and Phillips¹²² state that "the fusion temperature of the solder is often at least 180°F (82°C) below the fusion temperature of the work, although technicians expert in the art often join parts with solder possessing a fusion temperature of 90°F (32°C) or less below that of the work."

(The centigrade calculations are the authors' errata and should read 100°C and 50°C respectively.)

When soldering gold alloys the solder should be selected with a fusion range of at least 100°F (55.5°C) below the fusion temperature of the alloy being soldered.^{123,124,125} Ryge²³ recommends 100-150°F (55.5-83.3°C) and the American Dental Association¹²⁶ 100°C.

The variations expressed here would particularly relate to the soldering of a cast or wrought structure. The parent metal should not be heated near its melting range where grain growth occurs more readily. In wrought gold alloys grain growth occurs when the temperature of the metal is raised above the recrystallisation point and if it occurs during the soldering operation irreparable harm would be done.

The extent of the solder's melting range is also important. A narrow melting range permits the metal to suddenly liquify at the soldering temperature and quickly fill the joint. A wide melting range is used for building solders. Liquation¹²⁷ can occur with this type of solder. If the solder is held within its fusion range for too long a period the liquid portion may separate from the solid portion and become lost by capillary action in the joint, or by alloying with the basis metal, leaving a weakened solder structure. The composition of the solder and the gold alloy would be changed and the desired physical properties of the joint lost.

Consequently, solders with a wide fusion range must be handled with the utmost care.

A wide temperature variation exists between the melting range of the wrought platinised gold (M.R. 940°C - 960°C) and the 18K gold solder (M.R. 740°C - 755°C) used in this study. This combination is recommended by the manufacturer* with the intention of limiting the problems associated with recrystallisation of the wrought wire.

Figures 50 and 51 show a normal soldered joint. The gold wire (B) does not show a fibrous or banded grain structure owing to the manufacturer's recovery heat treatment and the soldering procedure, but the recrystallisation has progressed into grain growth. Figure 51A shows the structure of the gold wire prior to soldering.

Skinner and Phillips¹²⁸ state that it is possible for the grains in

* Precious Metal Refining Company.

the soldered part to act as nuclei of crystallisation for the solder and support this statement with a photomicrograph contributed by R.L. Coleman.²⁰

In figure 50 some areas at the interface show a grain boundary relationship between the solder and the gold alloy, but this relationship is more evident in all the other specimens examined. In figures 51, 52, 53 and 54 nucleation of the solder has been influenced by the grain structure of the gold alloy and epitaxy has occurred.

Figures 55 and 56 show the effect of excessive heat treatment with "diffusion" occurring along the grain boundaries as described by Ryge.²³ The grain boundaries are still visible in the gold solder and conform to those of the underlying gold alloy.

Skinner and Phillips¹²⁹ state that the structure of the grain boundaries varies from the rest of the grain. It is an area of transition between the differently orientated crystal lattices of the neighbouring grains. The fusion temperature is lower and not as definite as that of the grain. In figures 55 and 56 where the temperature and its time of application was increased, the grain boundaries of the alloy have widened. In the interface region the gold solder appears to have diffused or penetrated into the grain boundaries of the gold alloy. This would be enhanced by the observed apposition of the grain boundaries and their lower fusion temperature.

Recrystallisation of the wrought wire occurs when an increased temperature is used for soldering or the time of soldering is extended.

A soldered joint showing penetration and strong intergranular keying through united grain boundaries on microscopical examination possibly appears to be stronger than a joint formed by primary metallic bonding.

It has been shown that this is not the case and that a stronger joint with a less deleterious effect on the wrought basis metal is achieved at lower temperatures when both recrystallisation and intergranular penetration do not take place.^{15,23,30}

Inclusions.

The interfaces of figures 51, 52, 53 and 54 contain inclusions. After electrolytic etching these inclusions appeared on the interfaces.

In an attempt to remove these inclusions all the specimens were given additional ultrasonic cleaning by placing alternatively in distilled water and non-ammoniated detergent for five minute intervals, for a total of thirty minutes. The scratches on the specimens resulted from accidental surface contact during these procedures. The inclusions only occurred in the interface area and only involved the section towards the "hottest" part of the joint. No conclusion could be reached as to whether they were a function of intergranular breakdown, volatilization of base metals, inclusions from the solder or flux, or a by-product of the etching procedure. The specimens involved were refaced, polished and etched but the areas were still present. Metallic oxides have been reported in solder joints³⁰ and as inclusions in gold alloys¹³⁰ but no conclusion would be possible in this instance without additional investigation.

Comparison of the gold interfaces.

The difference between the gold soldered joint and the cast gold joint is primarily based on their functional requirements. Diffusion in gold soldered joints reduces the strength and increases the ductility of the wrought parent metal.^{15,23,30}

The electroformed gold matrix is specifically used to give an adaptable margin to the cast inlay. Consequently, the physical properties of the cast gold alloy are independent of the gold matrix. The margin of the pure gold matrix once annealed, does not respond to heat treatment unless strain hardening has occurred during its manipulation. The cast gold alloy can be heat treated to produce ideal physical properties as diffusion or recrystallisation, occurring within reasonable limits, as the metal interface will not adversely influence the gold matrix.

In soldering procedures a flux is essential to remove the surface contaminants. O'Brien et al.²⁴ found that the interfacial energies between gold solder and pure gold, or Type B gold alloy, indicate that the oxide layer does not play an important part in wetting when a flux is used. However, the use of a flux on the pure gold when casting a gold alloy against it caused a marked increase in porosity. This was probably due to the release and confining of the volatile constituents in the flux.

SUMMARY AND CONCLUSIONS

1. A technique for casting dental gold alloys to an electroformed gold matrix has been described. The "Thermal Expansion Technique" using a mould temperature of 700°C and a casting temperature of 20°C and 50°C above the liquidus of the alloy, and provision of a heat saturation time of five minutes before quenching gave an excellent union. Early evidence of diffusion is present.
2. The presence of the gold matrix in the investment did not interfere with the venting of the mould during the casting procedure.
3. The metallographic technique used gave a different interpretation to casting porosities as defined by Ryge et al. The incidence of microporosity due to dendritic shrinkage is greater than previously determined. By direct comparison of etching techniques some areas that would have been classified as pinhole porosity due to absorbed gases assumed a definite dendritic appearance (microporosity) when electrolytic polishing and etching procedures were used.
4. Subsurface porosity is discussed and presented as a gaseous porosity within the dendritic spaces instead of being dendritic shrinkage.
5. Residual carbon and by-products of electrodeposition were not evident at the interface of the two metals.

6. The use of a flux on the matrix is contraindicated due to an increase in the incidence of gaseous porosity.
7. Gold alloys as supplied by the manufacturers were investigated and it was found that granulated golds could contain gaseous inclusions.
8. Little difference was found between vacuum invested specimens cast on a Jelenko Thermotrol Casting Machine and vibrator invested flame melted, hand-swung inlays. Due to the advantages of a standardised technique, machine casting and vacuum investing is preferred.
9. The gold soldered joints showed an epitaxial relationship between the grain boundaries of the gold alloy and gold solder.
10. When diffusion occurred, the grain boundaries of the solder merged into the grain boundaries of the gold alloy.
11. Inclusions on the gold solder-gold alloy interface occurred. These may have been due to oxide formation, but no definite conclusion could be reached.

MATERIALS AND METHODS.

PART II. CHROMIUM CONTAINING ALLOYS.

MATERIALS AND METHODS.

PART II.

Chromium containing alloys.

a. Gold solder - cobalt-chromium alloy interface.

A quantity of cast cobalt-chromium alloys was obtained and sections approximately 1cm. long, 5mm. wide and 1mm. thick were cut. Flux and 18K gold solder was applied to these sections and they were heated over a Gruenburg burner until the solder flowed.

The cobalt-chromium alloys have the following composition:¹³¹

a. (Vitallium) Cr. 30 per cent

Co. 62.5 per cent

Fe. 1.0 per cent

C. 0.5 per cent

Si. 0.5 per cent

b. (Nobilium) Cr. 32 per cent

Co. 62 per cent

C. 0.35 per cent

Si. 0.35 per cent

Mo. 5.0 per cent

Mn. 0.69 per cent

B. $\leq$ 0.001 per cent

The flux used was Comweld Silver Brazing Flux No.2 and the only details available from the manufacturer state that it contained 31% potassium fluoborate and 28% potassium metaborate.

The 18K gold solder was the same as described in Part I.

Twenty solder sections were mounted edge wise in cold-curing acrylic resin and a connection for electrolytic polishing and etching was established as described for the gold specimens in section

Part 1.a.

Initial surfacing was carried out on the Lunn pregrinder followed by 7μ diamond paste. After washing and further polishing with a β diamond paste, final polishing of the cobalt-chromium was effected by using Struers RP3 electrolyte, with a small quantity of Microid Alumina added, at a current density of 0.3 amp./dm^2 for 2 minutes.

The structure of the cobalt-chromium was partially etched by using Struers A2 electrolyte on the Disa Electropol at a current density of 20 amp./dm^2 for 10 seconds.

Etching of the gold and cobalt-chromium was completed by use of the external etching apparatus of the Disa Electropol and a 10% potassium cyanide electrolyte. The current density was 10 amp./dm^2 and the specimens were etched for 5 seconds, rinsed dried and examined under the microscope. This procedure was repeated until a satisfactory etch was produced - usually 15 to 25 seconds of anodic etching was required.

b. Electroplated gold - cobalt-chromium alloy interface.

Several cast cobalt-chromium dentures were obtained and gold plated as described by the author.⁹¹ Twenty specimens approximately 1cm. long, 5mm. wide and 1mm. thick were cut from appropriate areas

of the dentures. These specimens were mounted, polished and etched as previously described in section Part II.a.

c. Silver solder - stainless steel interface.

A quantity of 16 gauge 321 austenitic stainless steel was purchased. Pieces 1cm. long, and 5mm. wide were cut from the strip. Two pieces of stainless steel with flux and silver solder between them were heated with a gas-air torch until the solder flowed and an effective sandwich union was achieved.

The flux used was Comweld Silver Brazing Flux No.2, as previously described and the silver solder was Easy Flow No.2 which has the following composition and properties:

Silver	40 per cent
Copper	20 per cent
Zinc	15 per cent
Cadmium	25 per cent
Melting range.	608°-617°C
Tensile strength.	30 tons/sq. inch
Elongation %.	30
Hardness.	131 Vickers

Twenty specimens were mounted edgewise and an electrical contact attached as described in section Part I.a.

Due to the difficulty in maintaining the surfaces of the silver solder and the stainless steel in the one plane during the polishing and etching

procedures it was reasoned that the most satisfactory method would be to use a sandwich type joint.

Initial surfacing and polishing were as described in section Part I.a.

The silver solder only was etched in 10% ammonium persulphate solution using the external etching apparatus of the Disa Electropol and applying a current density of 8 amp./dm^2 for 5 seconds.

The specimen was washed and dried in a stream of oxygen to assist the passivation of the stainless steel. After allowing a further interval of time to ensure passivation of the stainless steel surface a flash coating of gold was applied over the entire specimen utilising the electrolyte described in Part I.a. and applying a current density of 3.2 amp./dm^2 for 10 seconds.

The estimated thickness of this deposit was 0.2microns.
(8×10^{-6} in).

The non-adherent gold was removed from the stainless steel by lightly polishing with $\frac{1}{3}$ diamond paste.

The exposed stainless steel was then electrolytically etched by using the Disa Electropol and Struers A2 electrolyte at a current density of 20 amp./dm^2 . The specimen was then placed on the Realopol using Struers RP3 electrolyte with Microid Alumina to remove the gold layer. A current density of 45 ma./dm^2 was used and the specimen examined every five seconds until levelling of the interface surface occurred. A period of 10-15 seconds was required for this part of the procedure. Any minute trace of gold that remained on the silver solder would have

the same structure as the basis metal.¹³²

Electron microprobe analysis of the stainless steel, silver solder interface.

The specimen that provided figures 65 and 66 was examined with an electron microprobe analyser by Mr. F. Scott of the University of N.S.W. The resolution of the probe was approximately $2-3\mu$ and manual scanning as well as an autographic result was carried out.

d. Gold solder-stainless steel interface.

Twenty sandwich specimens were prepared as described in Part II.c. with the exception that gold solder, of the composition and properties described in Part I.b. was substituted for silver solder.

The specimens were mounted and polished as previously described in Part I.a.

The stainless steel was depassivated by placing in 10% H_2SO_4 for 1 minute and then etched using the Disa Electropol and Struers electrolyte A2 with an applied current density of 20 amp./dm^2 for fifteen seconds. This only partially etched the gold solder and the final etching of the solder was accomplished by using 10% potassium cyanide at a current density of 10 amp./dm^2 for 15 seconds in the external etching apparatus of the Disa Electropol.

e. Electroplated gold - stainless steel interface.

Stainless steel strips of the size and composition previously

described were gold plated using the same technique, electrolytes and conditions as described for cobalt-chromium alloys. The gold plated stainless steel was then clad in electroplated nickel to protect the soft gold plating. Approximately 1.5mm. of nickel was deposited by applying a current density of 4.3 amp./dm^2 for 30 hours using Watt's electrolyte of the following formula:

nickel sulphate	210g
nickel chloride	60g
boric acid	30g
water to 1 litre	

The mounting, polishing and etching procedures were the same as previously described in Part II.a., twenty specimens being prepared for examination.

f. Resistance welded stainless steel interface.

Two experienced welders were asked to produce welded joints as encountered in orthodontic practice and these were examined as examples of autogenous bonding.

Operator A produced eight specimens of spot welded, lapped, Unitek Resilient Flatwire (0.015 X 0.028 in.) using a Unitek 1050 Multi-purpose Welder.

The only information available on this wire stated that it was composed of Unitek Permachrome metal that had been subjected to a four hour heating cycle for stress relief in a hydrogen atmosphere.

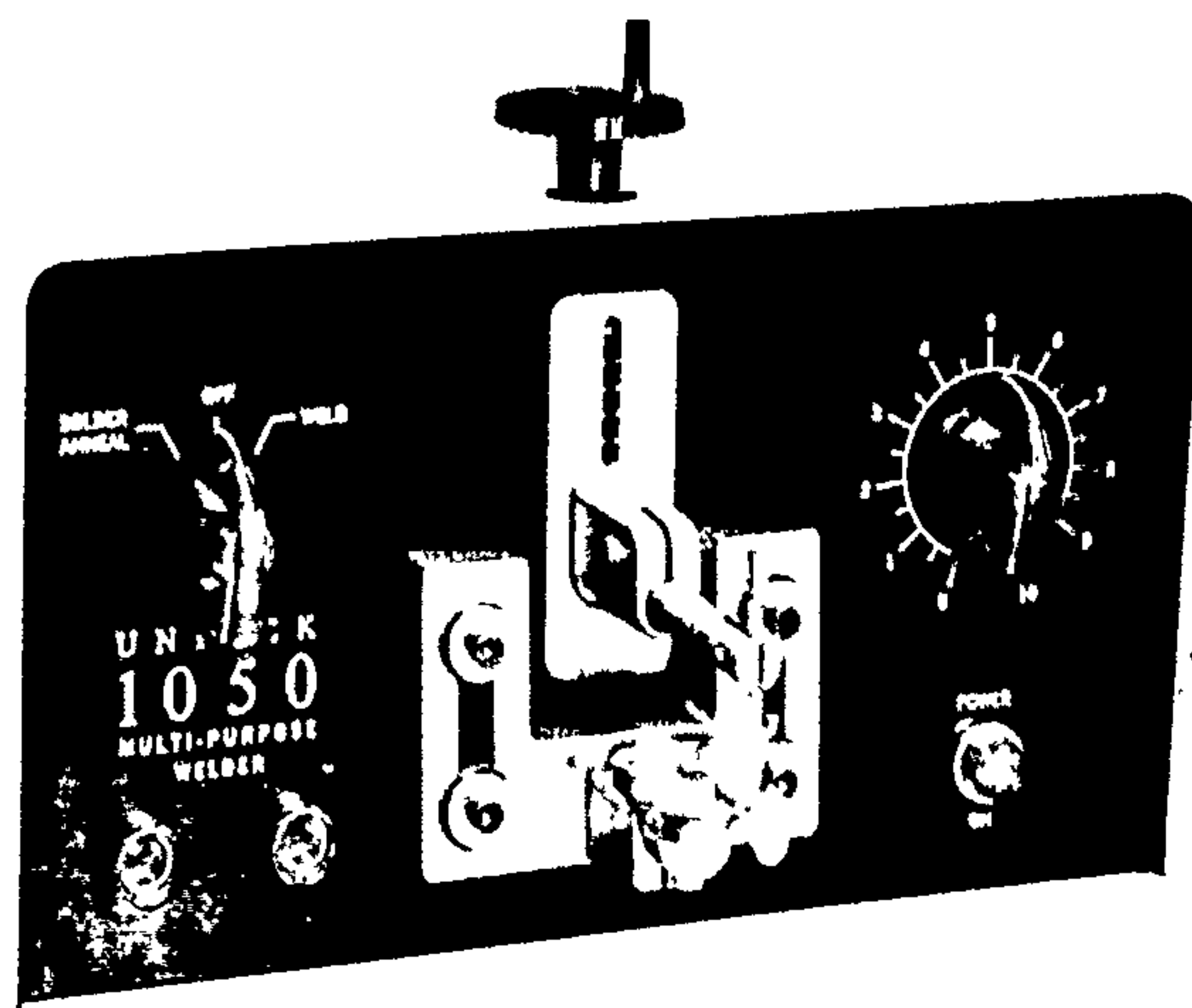


Figure 57. Unitek 1050 Multi-purpose Welder.

The Unitek 1050 Multi-purpose Welder (Fig. 57) is a capacitance welder that operates on 115 volts, 60 cycle AC. The welding impulse is controlled by the selector (A) and control settings of 4, 6, 7, 9 and 10 units were used.

Operator B produced twelve specimens using a Rocky Mountain 506 Welder. Six specimens were prepared using Rocky Mountain 0.005in. Chrome Alloy band material with two Broussard auxiliary edgewire brackets welded on to each specimen, and six specimens were prepared using lapped Rocky Mountain 0.005in. band material.

The Rocky Mountain Chrome Alloy is described as a stabilised 18/8 type 302 stainless steel.

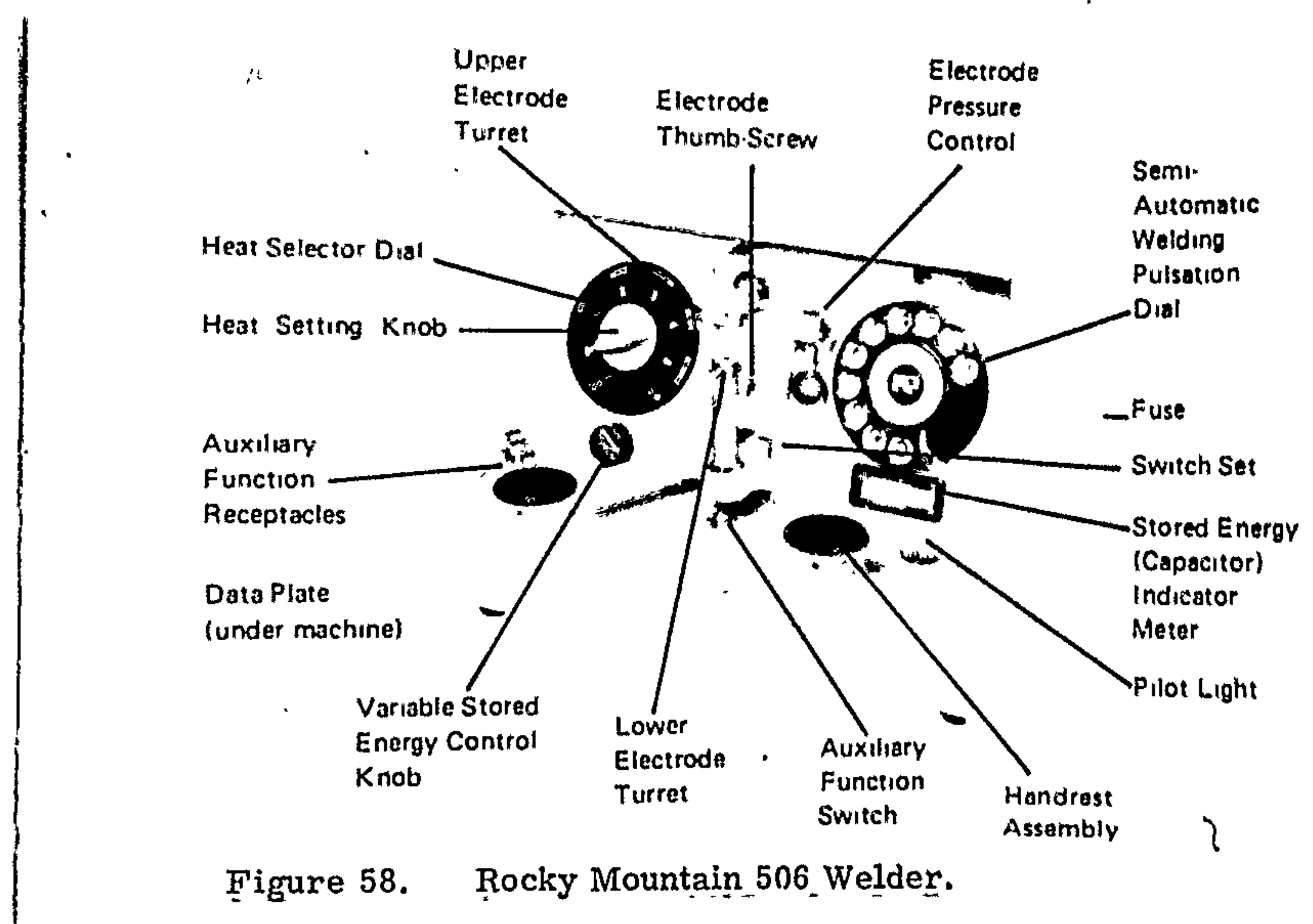


Figure 58. Rocky Mountain 506 Welder.

The Rocky Mountain 506 Welder (Fig. 58) has a one capacitor heat and a 4-heat transformer circuitry that operates on 240 volts, 50 cycle AC.

Three specimens of each series were made with the heat selector on 3 units (medium) and pulsations of 2, 4 and 6.

A further series of specimens with similar pulsations were prepared with the heat selector on 4 units (heavy).

The specimens were mounted and prepared for metallographic examination as described in Part I.a. and electrolytically polished on the Disa Electropol using a current density of 27 ma./dm^2 for 10 seconds and Struers A2 electrolyte.

The external etching apparatus was used to etch the electropolished specimens in an electrolyte of 10% ammonium persulphate at a current density of 15 ma./dm^2 applied for 15 - 20 seconds.

RESULTS.

PART II. CHROMIUM CONTAINING ALLOYS.

RESULTS.

PART II. Chromium containing alloys.

a. Gold solder - cobalt-chromium alloy interface. (Figures 59, 60 X600).

All these experiments showed close affinity between the gold solder and the cobalt-chromium. At the interface the gold solder accurately followed the smooth, straight surface of the cobalt-chromium alloy and there was no evidence of interalloying.

Figure 59 illustrates the Vitallium, gold interface and figure 60 the Nobileum, gold interface.

b. Electrodeposited gold - cobalt-chromium alloy interface. (Figures 61, 62, 63 and 64 X600).

The anodic etching of the cobalt-chromium alloy during the electroplating procedure produced an irregular interface. This varied from a slight effect, as seen in one specimen (figure 63) to the general overall etching effect as seen in figures 61, 62 and 64. The electrodeposited gold closely followed the conformation of the cobalt-chromium and the irregular surface could provide it with additional keying for anchorage. All specimens showed a line of current interruption in the electrodeposited gold owing to the castings being removed from the electrolyte for inspection during the electroplating procedure.

Figure 61 shows a Vitallium alloy, gold interface and figures 62, 63 and 64 show Nobileum alloy, gold interface.

c. Silver solder - stainless steel interface. (Figures 65 X500, 66 and 67 X800, 68 X1200).

These specimens showed a relationship between the lamellar structure of the silver solder and the notched grain boundaries of the stainless steel interface. Whilst there was no direct evidence of inter-alloying, the surface of the stainless steel had influenced the nucleation of the silver solder and the solder had penetrated into the notched intergranular areas.

Electron microprobe analysis of the stainless steel, silver solder interface. (Figure 69.)

This revealed no evidence of alloying within the resolution of the instrument.

The autographic area covered 50μ but longer manual scans confirmed the results indicated in the autographic scan.

The slight discrepancy noted in the "X" axis of the autographic recording between the elements analysed was due to the deflection of the electron beam over the specimen and defocussing of the spectrometer - this is an inherent instrumental error.

d. Gold solder - stainless steel interface. (Figures 70 and 71 X600).

The gold solder showed a smaller grain structure at the stainless steel interface and epitaxy occurred across the metal junction. In figure 71 the gold solder does not conform to the surface irregularities of the stainless steel and notching of the grain boundaries of the stainless steel has occurred. This effect was observed in most specimens.

e. Electroplated gold - stainless steel interface. (Figures 72, 73 and 74 X600).

The union was not as tight as that of the electroplated gold, cobalt-chromium series (Part II.b.). After the initial gold plating had been completed the current density was increased excessively leading to a "burnt" deposit and grain separation. At the interface the gold showed many points of nucleation and a small grain size due to rapid deposition. This would also have been accentuated by the disturbed surface layer produced by the activation procedure and the very thin protective nickel surface.

The interface of the gold does not closely follow the stainless steel interface. This could be due to the levelling effect of the nickel surface or a microporous surface, caused by the etching procedure. The high chloride content of the Woods' nickel electrolyte during surface activation, when the stainless steel is anodic, could have caused preferential dissolution of the iron, chromium, and nickel content of the stainless steel.¹³³

f. Resistance welded stainless steel interface. (Figures 75, 76, 77 X150 and 78, 79, 80, 81, 82 X300).

The specimens produced by the Unitek 1050 Multi-purpose Welder varied from inadequate bonding of the resilient flat wire (Settings 4 and 6) to excessive heat formation (Settings 9 and 10) characterised by large nugget formation, excessive indentation and burning of the wire surface, expulsion of molten metal, and destruction of the physical properties of the wire.

A satisfactory bond is shown in figure 75. The weld nugget (B) forms an autogenous joint between the two stainless steel wires and the fibrous structure of the wire has not been destroyed beyond the weld nugget except for the small areas showing burning at the surface of the wire.

Figures 76 and 77 show complete destruction of the stainless steel wire and its replacement by a cast structure with inferior physical properties. The surfaces of the wire show excessive indentation and expulsion of the molten metal has occurred.

The specimens produced by the Rocky Mountain 660 Welder showed a series of fusion areas or nuggets at the metal interface due to the multi-pulse procedure.

Figures 78 and 79 show specimens produced on a heat regulator setting of 3 (medium). Two weld nuggets are clearly visible in both figures. The weld nuggets show a typical radially grained structure and porosity has occurred in figure 79.

Figures 80, 81 and 82 show specimens produced on a heat regulator setting of 4 (heavy). All the specimens display multi-nugget formation and areas of porosity. The grain structure of the stainless steel beyond the weld area has not been altered.

Figure 59.

Gold solder, cobalt-chromium interface (Vitallium) X 600.

Legend.

- A. 18K gold solder
- B. Cobalt-chromium (Vitallium)
- C. Interface showing close affinity between gold and cobalt-chromium.

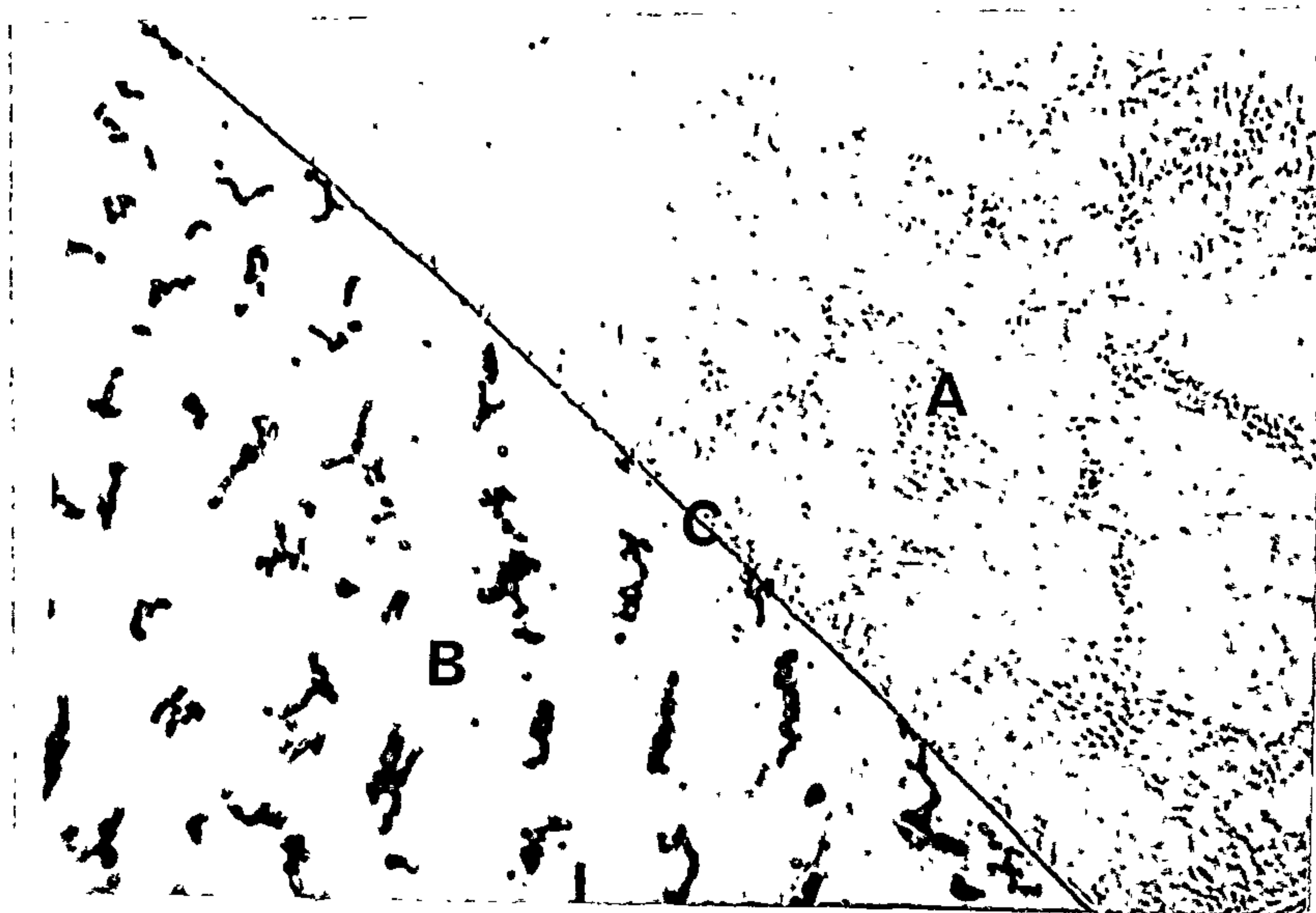




Figure 60.

Gold solder, cobalt-chromium interface (Nobilium) X 600.

Legend.

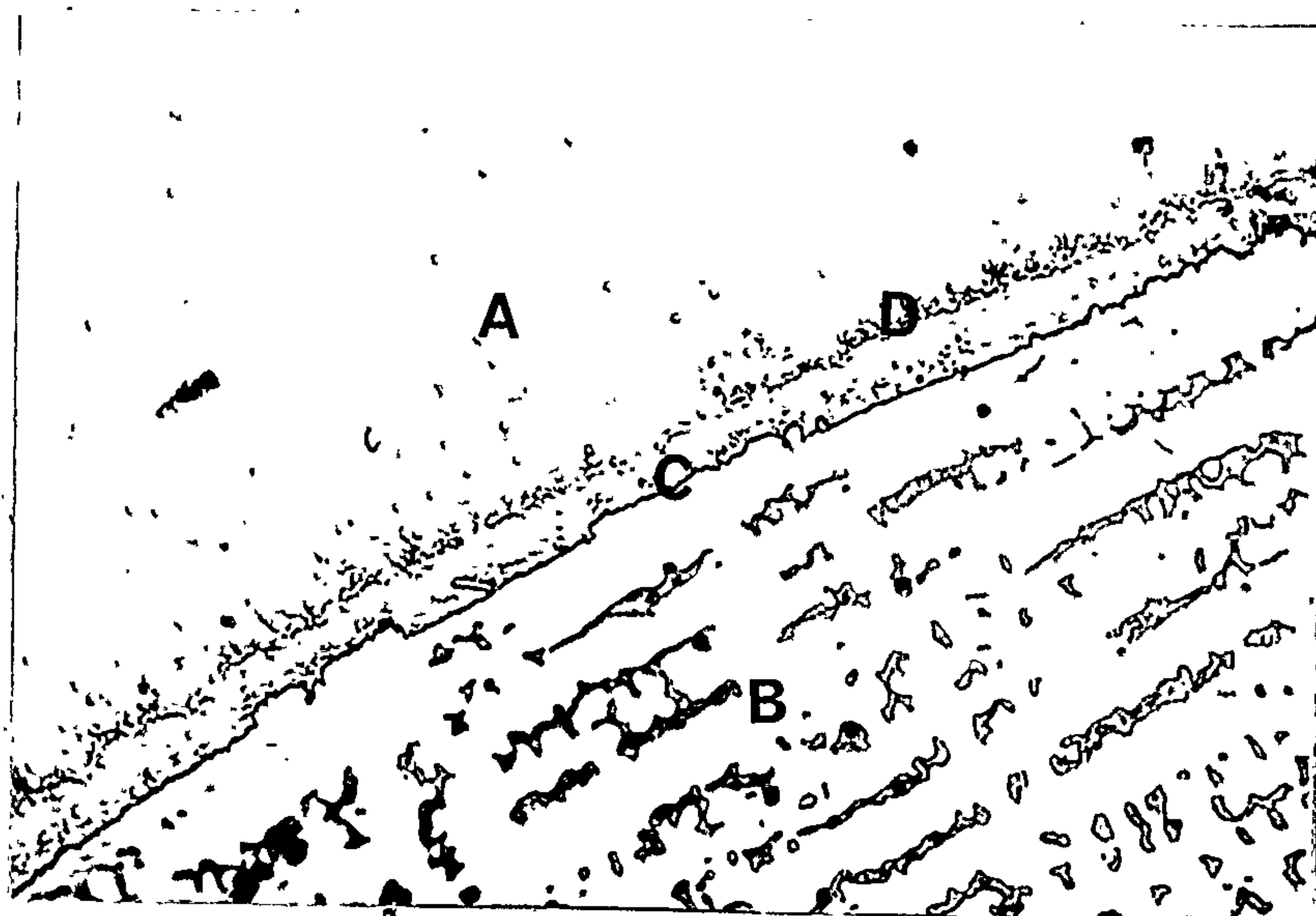
- A. 18K gold solder
- B. Cobalt-chromium (Nobilium) exhibiting a greater number of carbide islands than the Vitallium alloy.
- C. Interface shows close affinity between gold and cobalt-chromium.

Figure 61.

Electrodeposited gold, cobalt-chromium interface X 600.

Legend.

- A. Electrodeposited gold
- B. Cobalt-chromium (Vitallium)
- C. Irregular interface
- D. Line due to current interruption when electrodepositing gold.



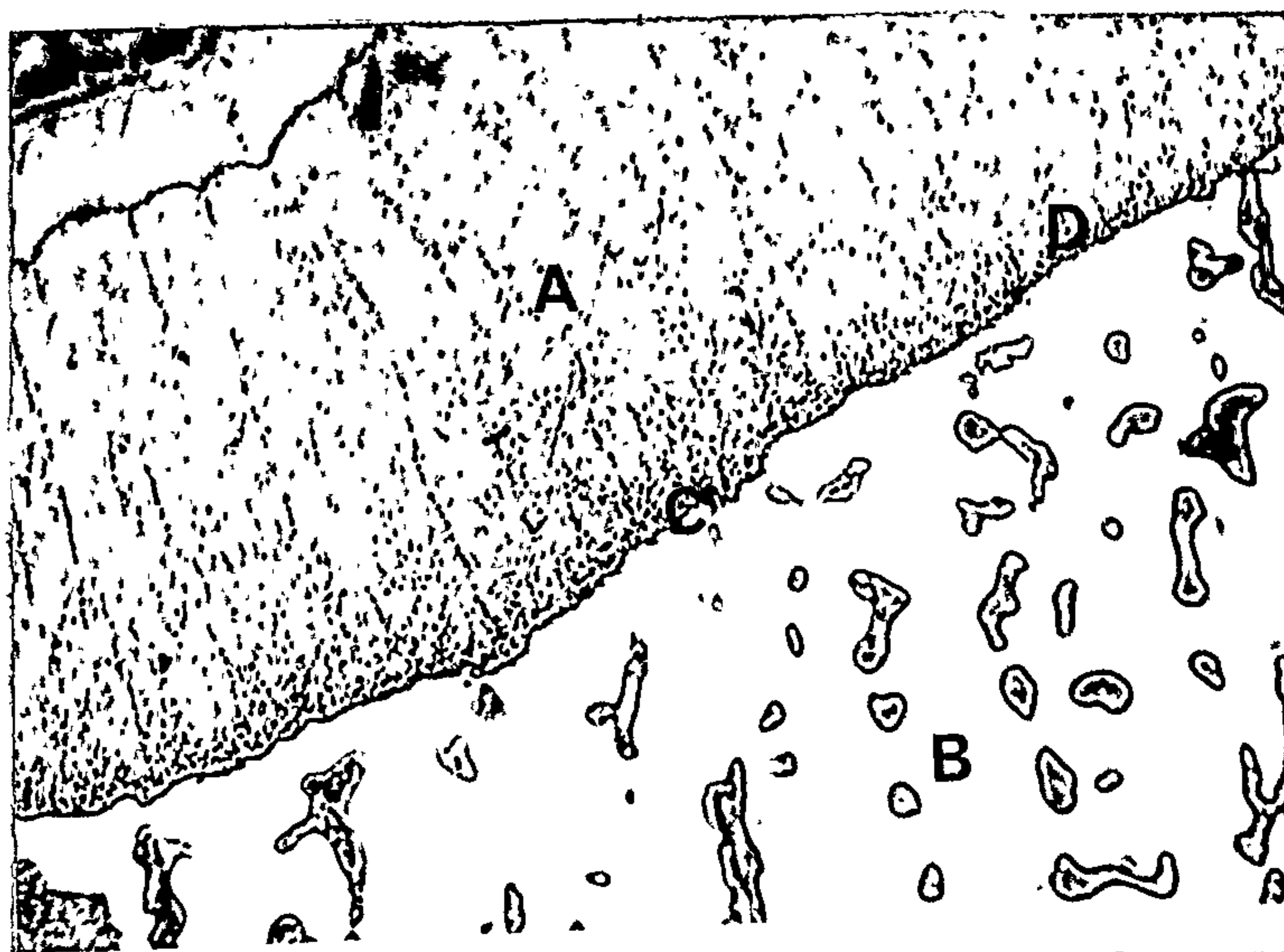


Figure 62.

Electrodeposited gold, cobalt-chromium interface X 600.

Legend.

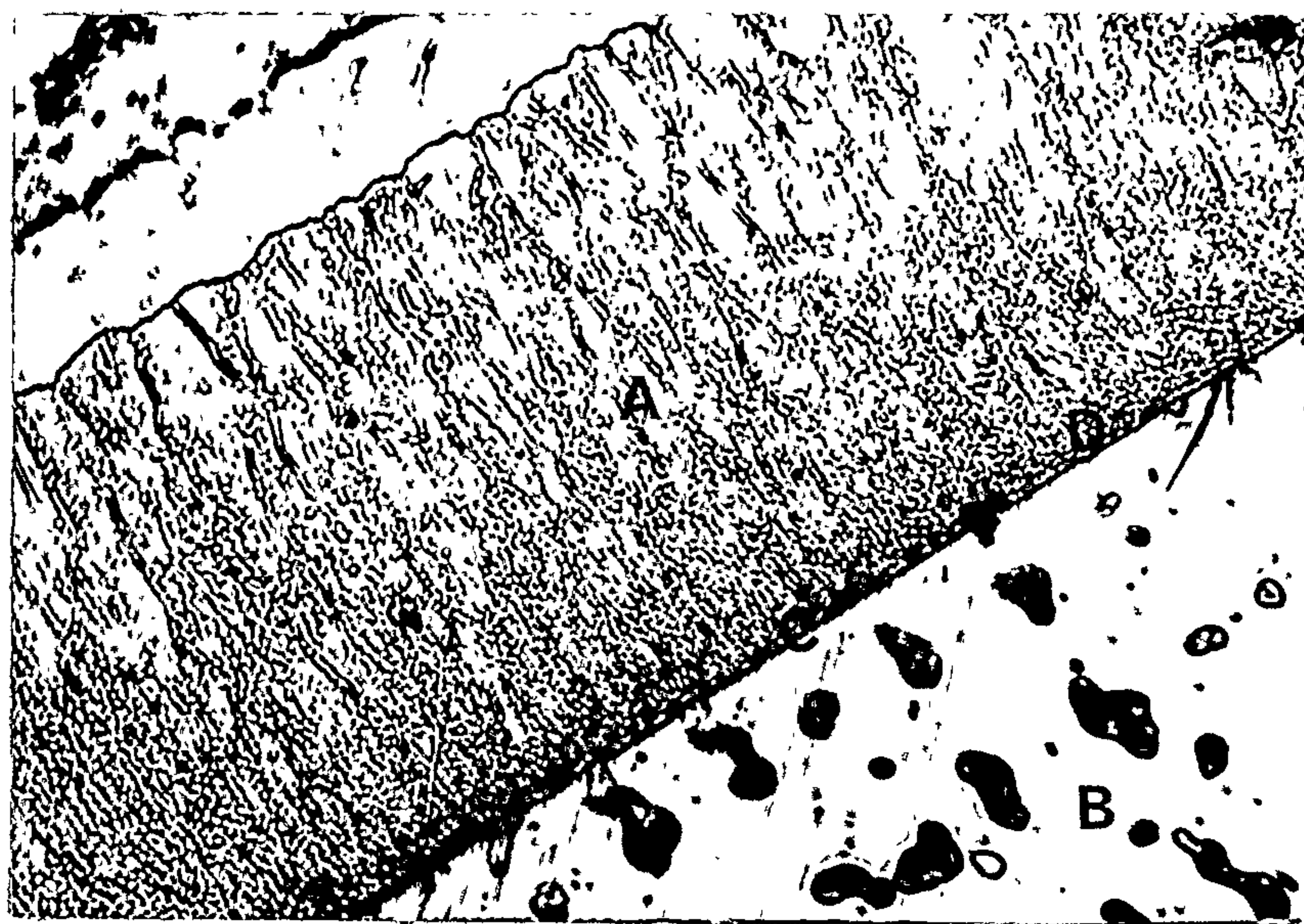
- A. Electrodeposited gold
- B. Cobalt-chromium (Nobilium)
- C. Irregular interface
- D. Line due to current interruption when electroplating gold.

Figure 63.

Electrodeposited gold, cobalt-chromium interface X 600.

Legend.

- A. Electrodeposited gold
- B. Cobalt-chromium (Nobilium)
- C. Irregular interface
- D. Line due to current interruption when electroplating gold.



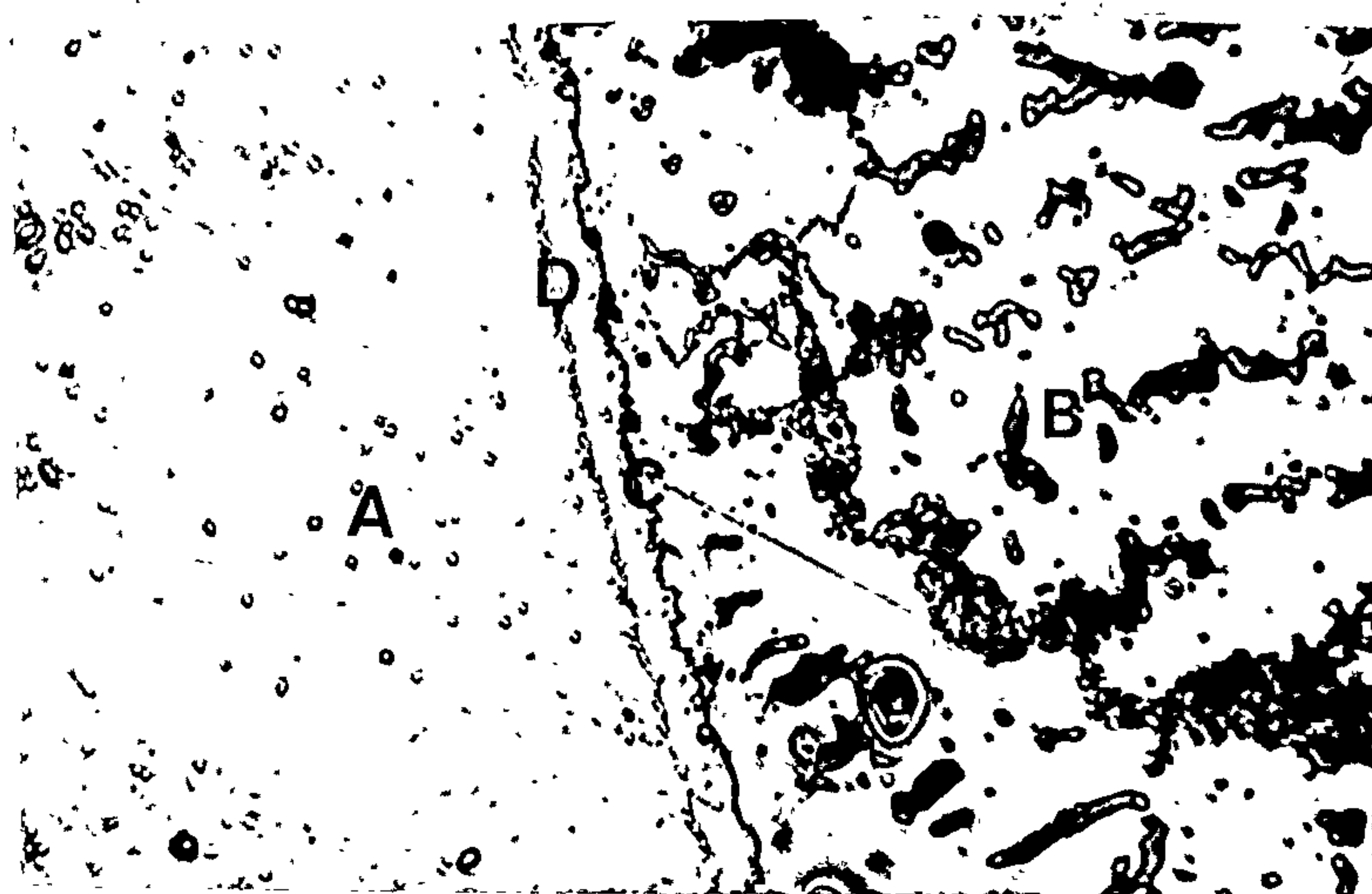


Figure 64.

Electrodeposited gold, cobalt-chromium interface X 600.

Legend.

- A. Electrodeposited gold
- B. Cobalt-chromium (Nobilium)
- C. Irregular interface
- D. Line due to current interruption when electroplating gold.

Figure 65.

Silver solder, stainless steel interface X 500.

Legend.

- A. Silver solder
- B. Stainless steel
- C. Interface.



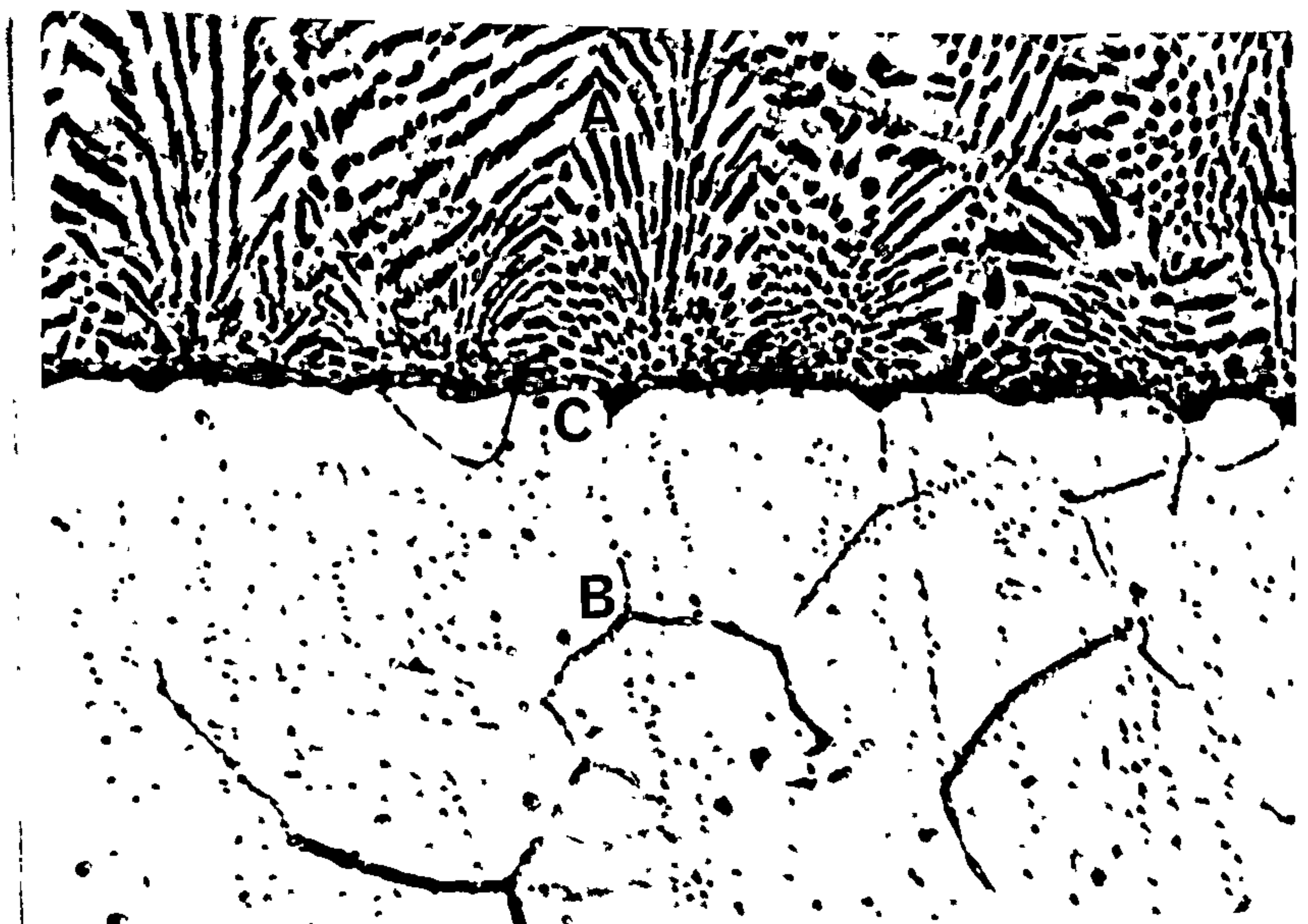


Figure 66.

Silver solder, stainless steel interface X 800.

Legend.

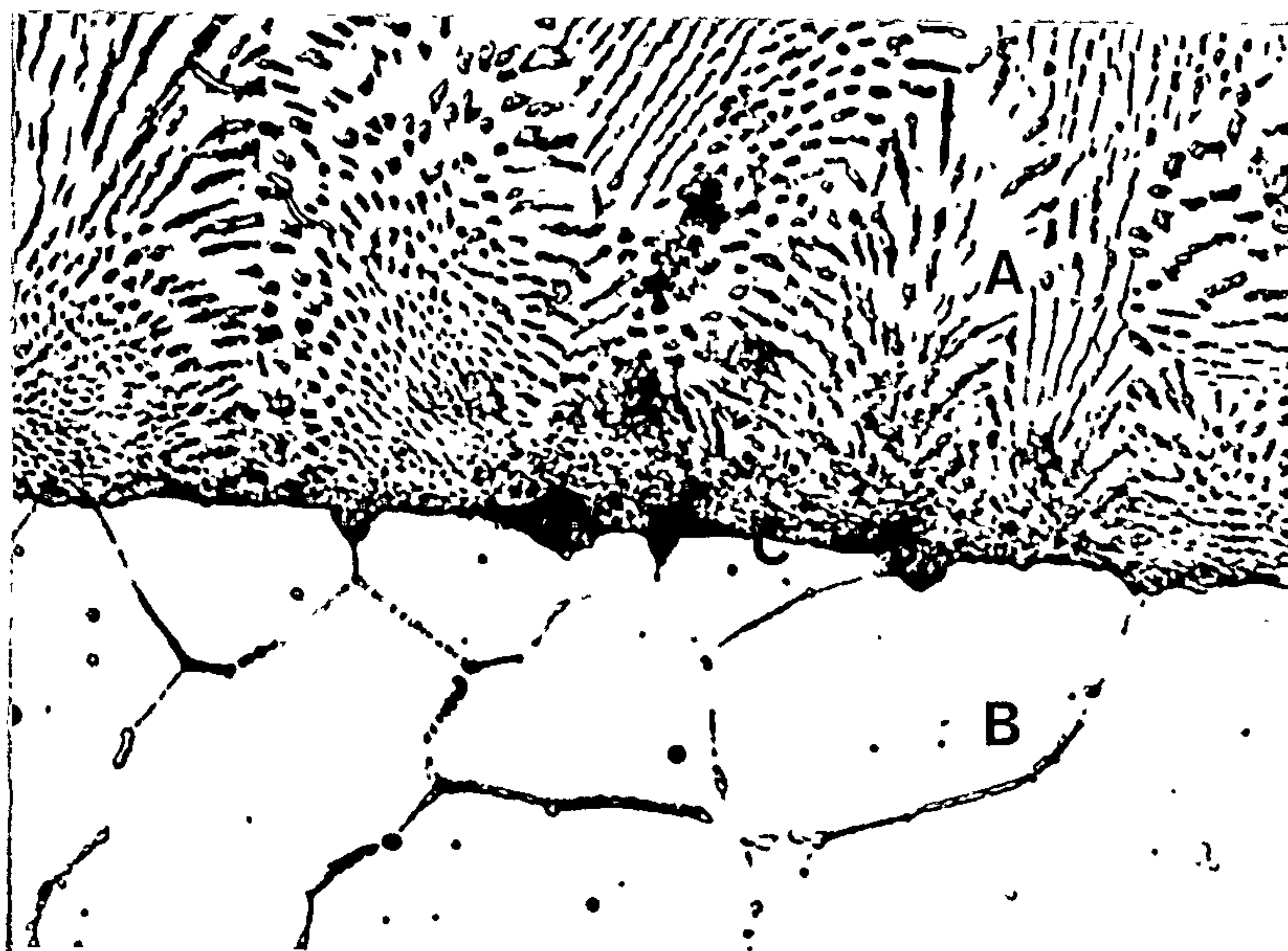
- A. Silver solder
- B. Stainless steel
- C. Interface - note the notched surface particularly at the grain boundaries of the stainless steel.

Figure 67.

Silver solder, stainless steel interface X 800.

Legend.

- A. Silver solder
- B. Stainless steel
- C. Interface.



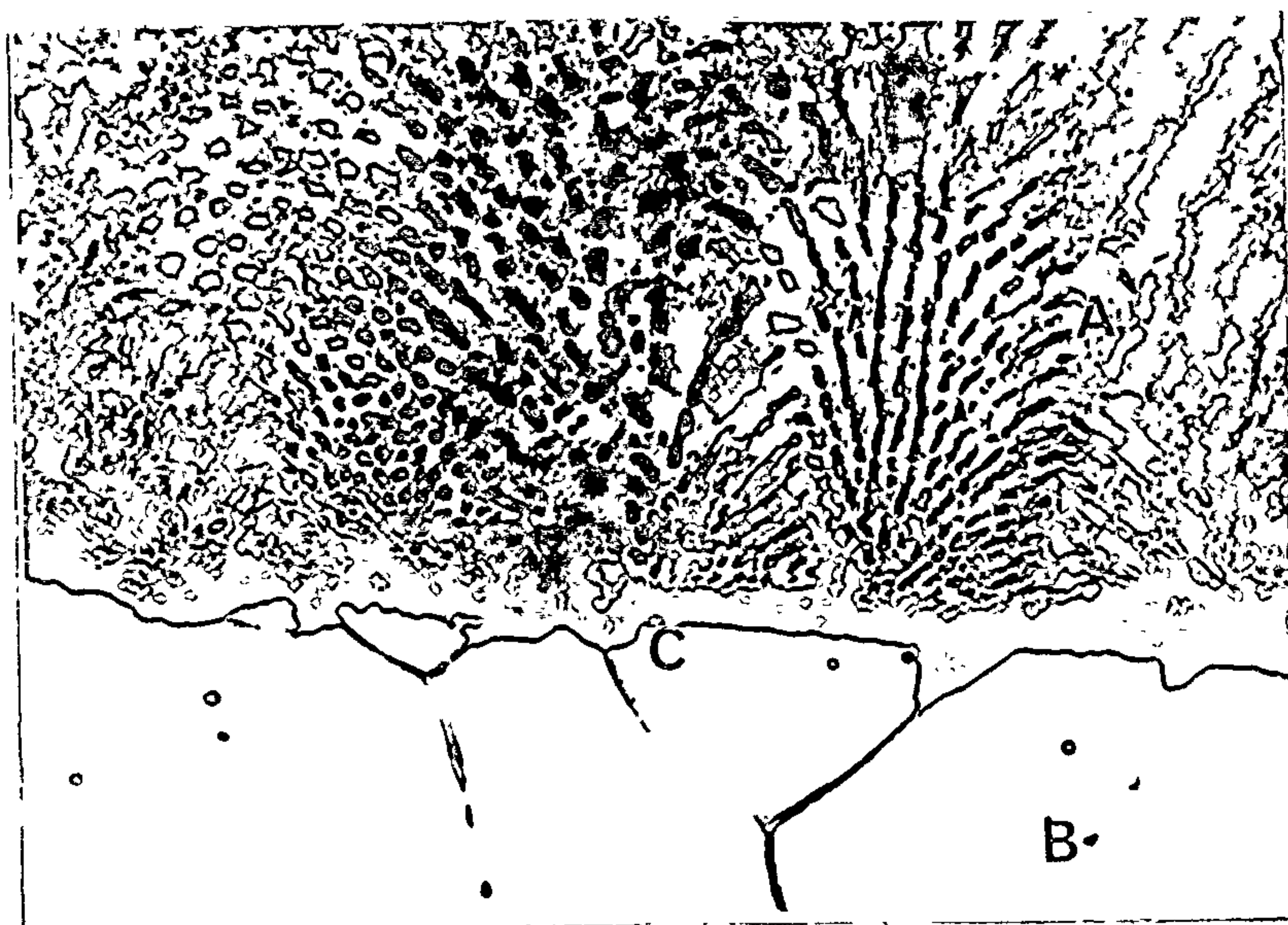


Figure 68.

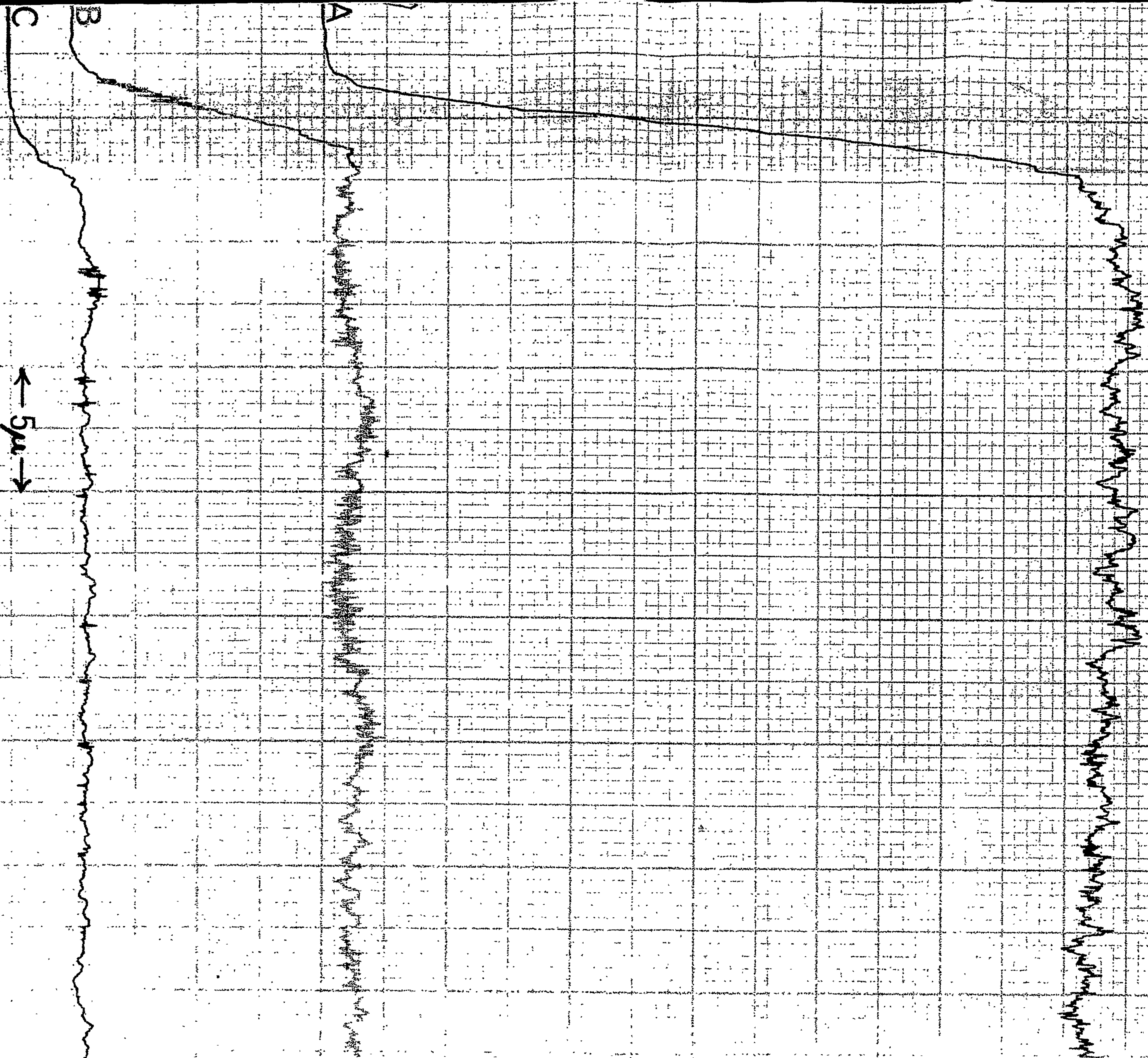
Silver solder, stainless steel interface X 1200.

Legend.

- A. Silver solder
- B. Stainless steel
- C. Interface.

Ag. Interface.
Sol.

Stainless Steel.



Legend.

A. Cr. base line.

B. Ni. base line.

Figure 69.

150.

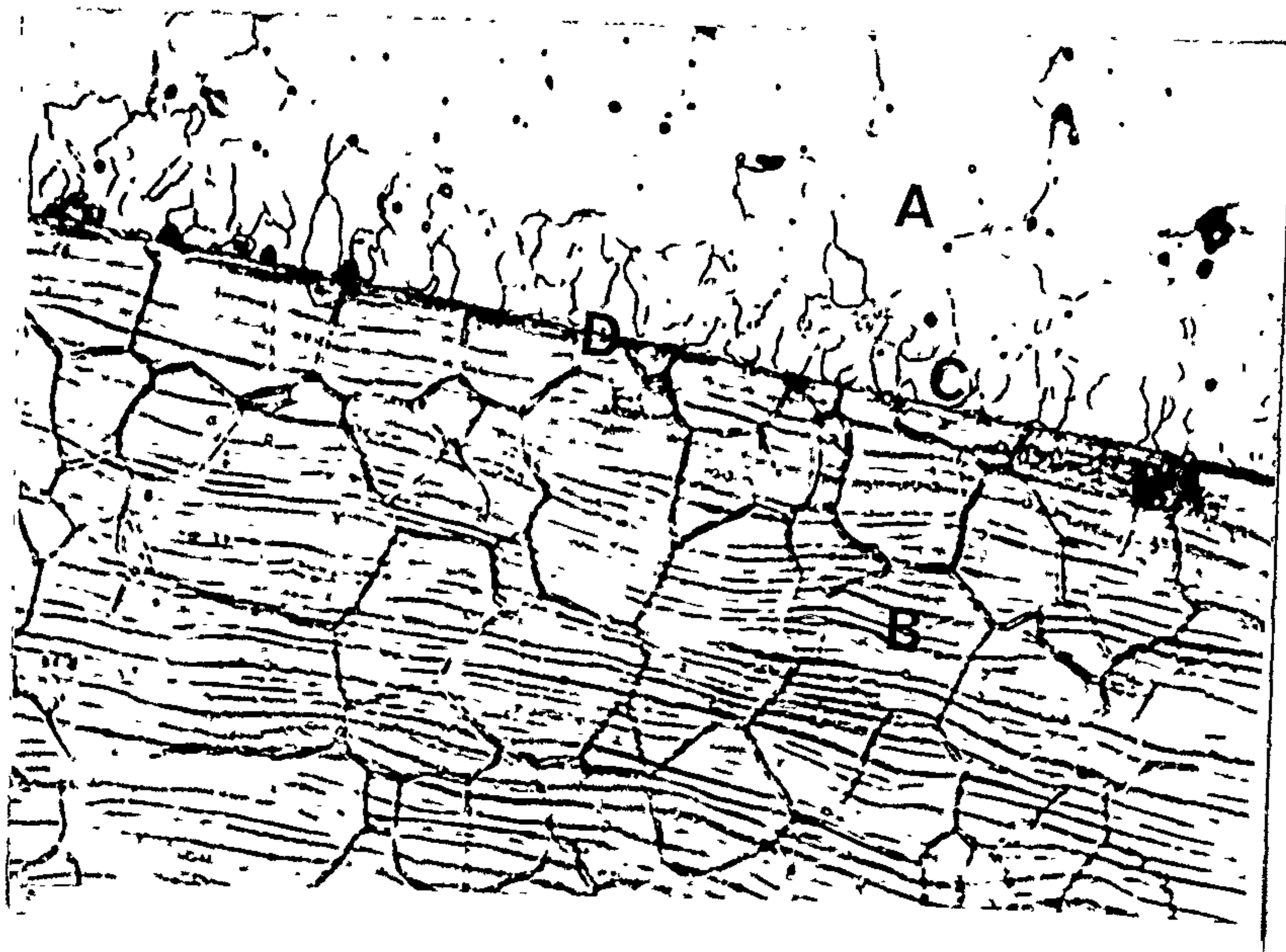
Results of the electron microprobe analysis.

Figure 70.

Gold solder, stainless steel interface X 600.

Legend.

- A. Gold solder
- B. Stainless steel showing fibrous banded structure and recrystallisation
- C. Interface - note small grain structure of gold at the interface
- D. Epitaxy.



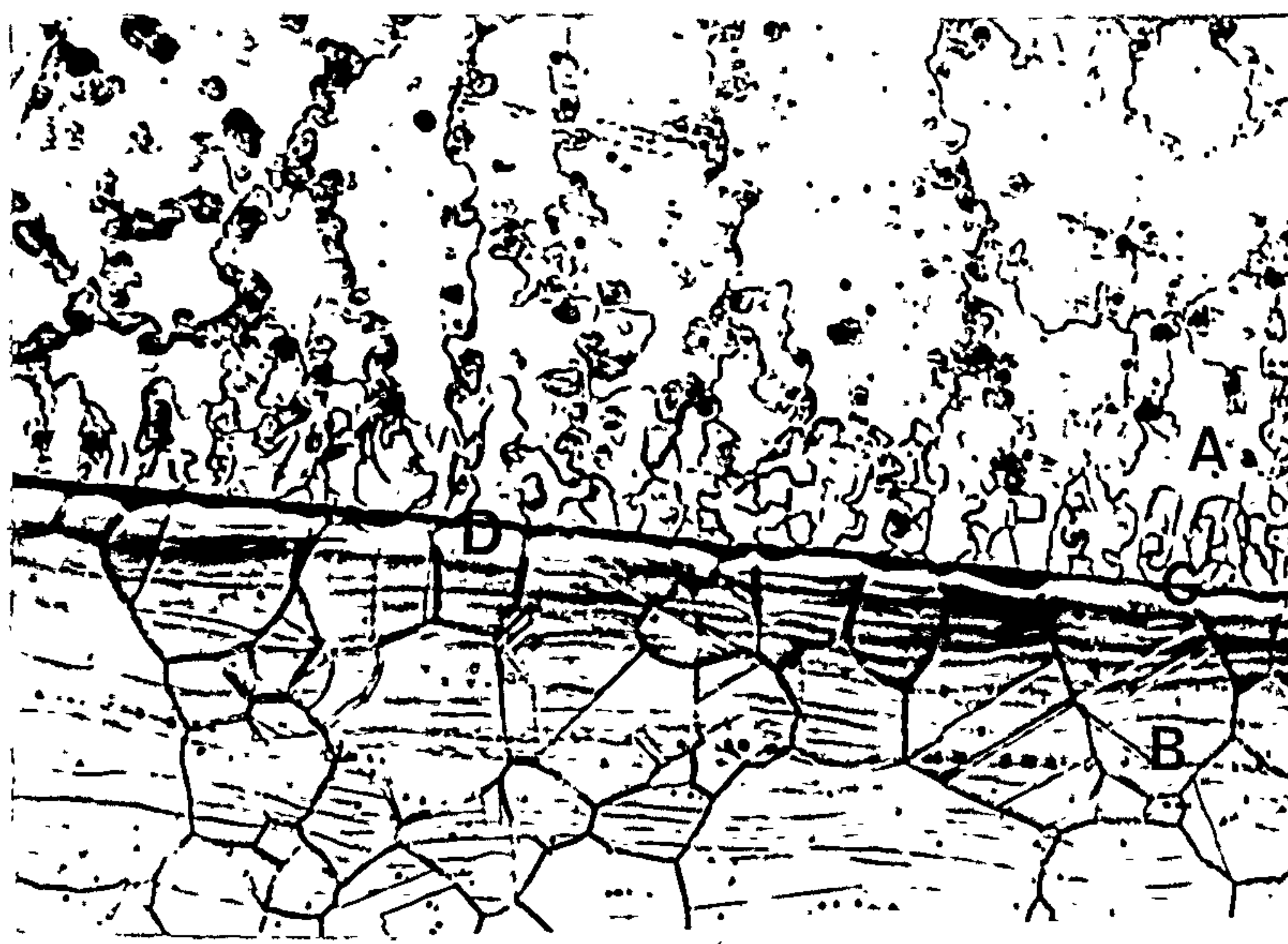


Figure 71.

Gold solder, stainless steel interface X 600.

Legend.

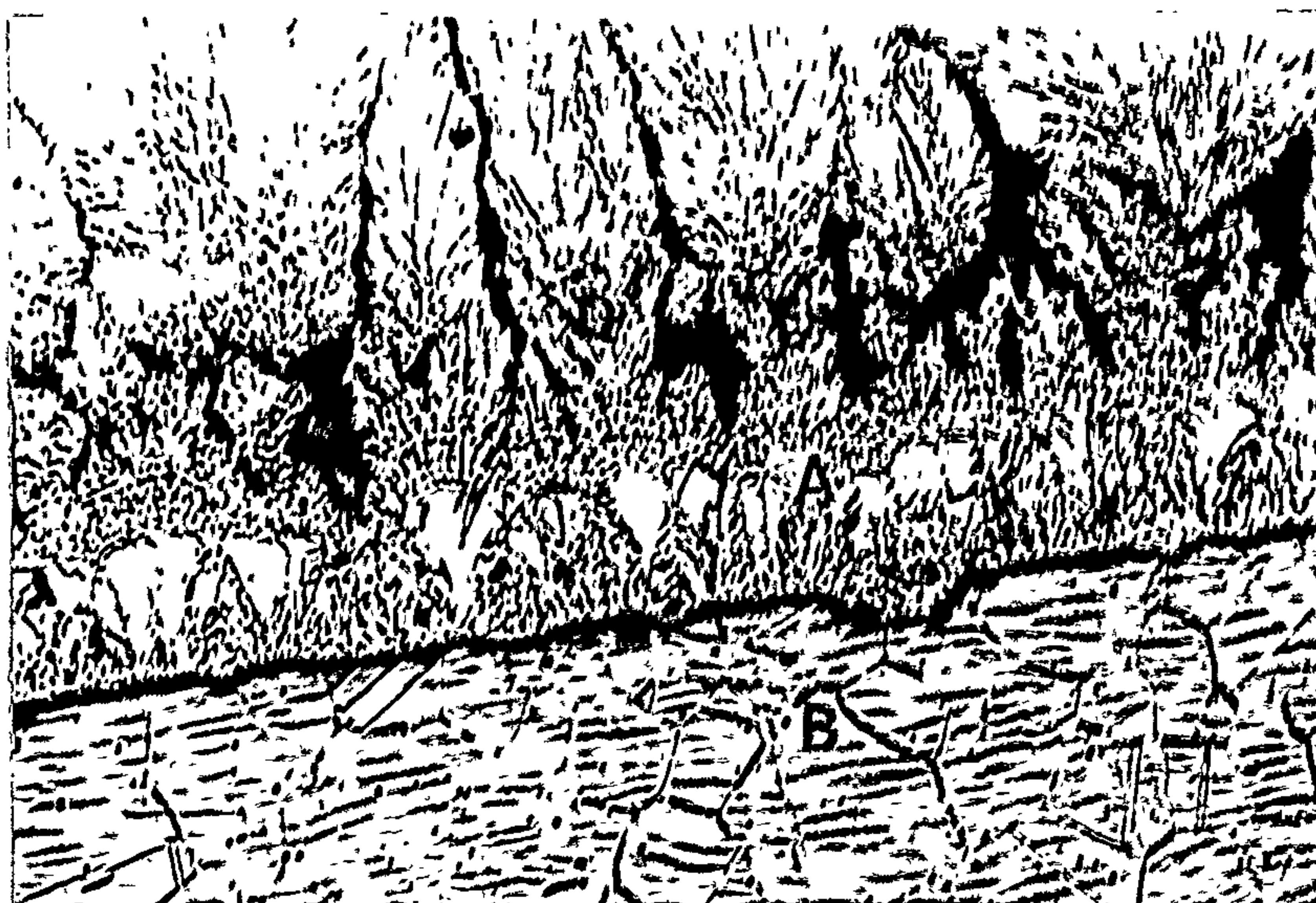
- A. Gold solder
- B. Stainless steel showing a fibrous banded structure and recrystallisation
- C. Interface - the gold solder does not conform to the surface irregularities of the stainless steel and the grain boundaries at the stainless steel interface have a "notched" appearance
- D. Epitaxy.

Figure 72.

Electroplated gold, stainless steel interface X 600.

Legend.

- A. Electrodeposited gold
- B. Stainless steel showing a banded, fibrous structure and recrystallisation
- C. Interface
- D. Burnt deposit of electrodeposited gold due to increased current density.



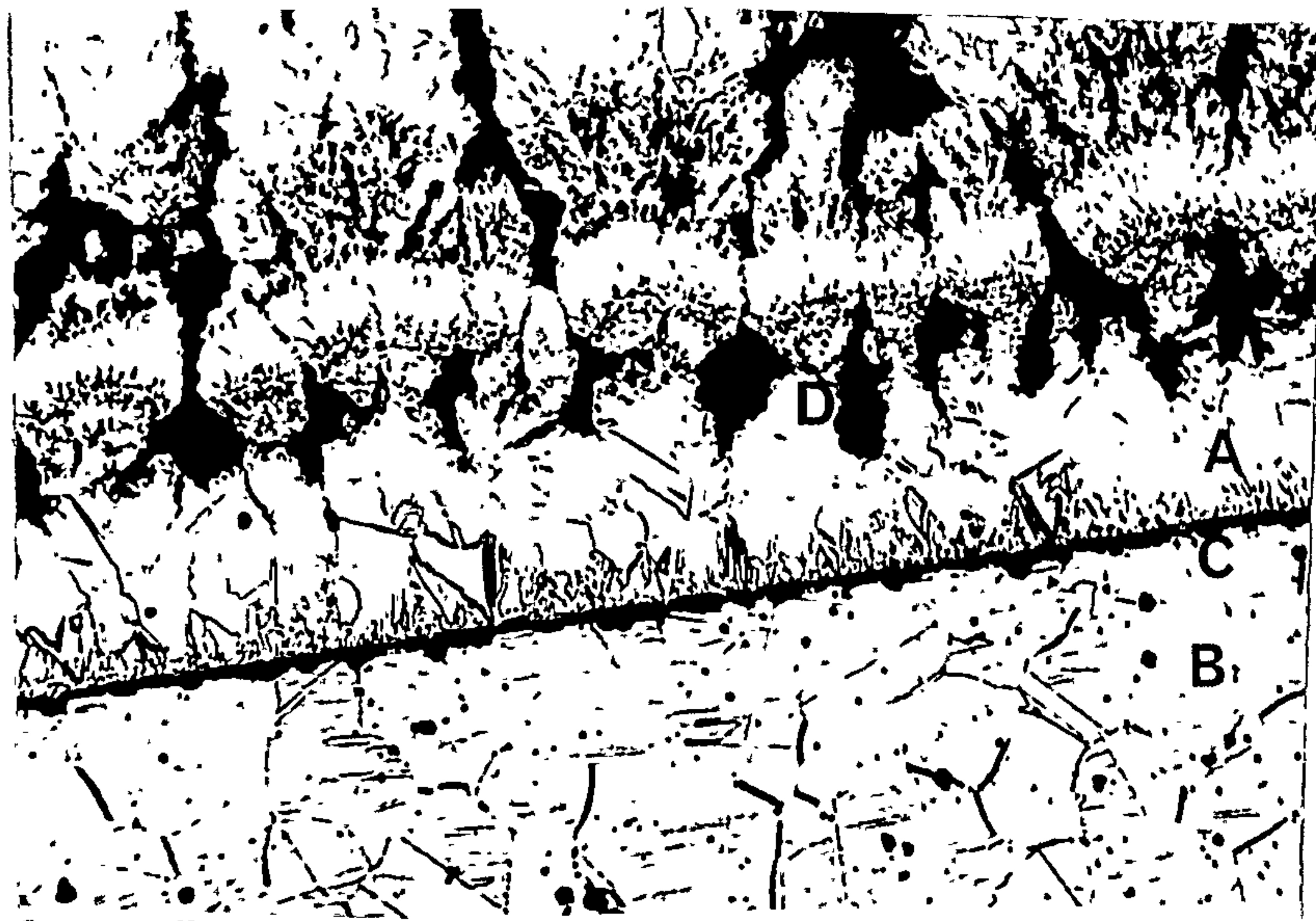


Figure 73.

Electroplated gold, stainless steel interface X 600.

Legend.

- A. Electrodeposited gold
- B. Stainless steel showing a banded, fibrous structure and recrystallisation
- C. Interface
- D. Burnt deposit of electrodeposited gold due to increased current density.

Figure 74.

Electroplated gold, stainless steel interface X 600.

Legend.

- A. Electrodeposited gold
- B. Stainless steel showing a banded, fibrous structure and recrystallisation
- C. Interface
- D. Burnt deposit of electrodeposited gold due to increased current density.



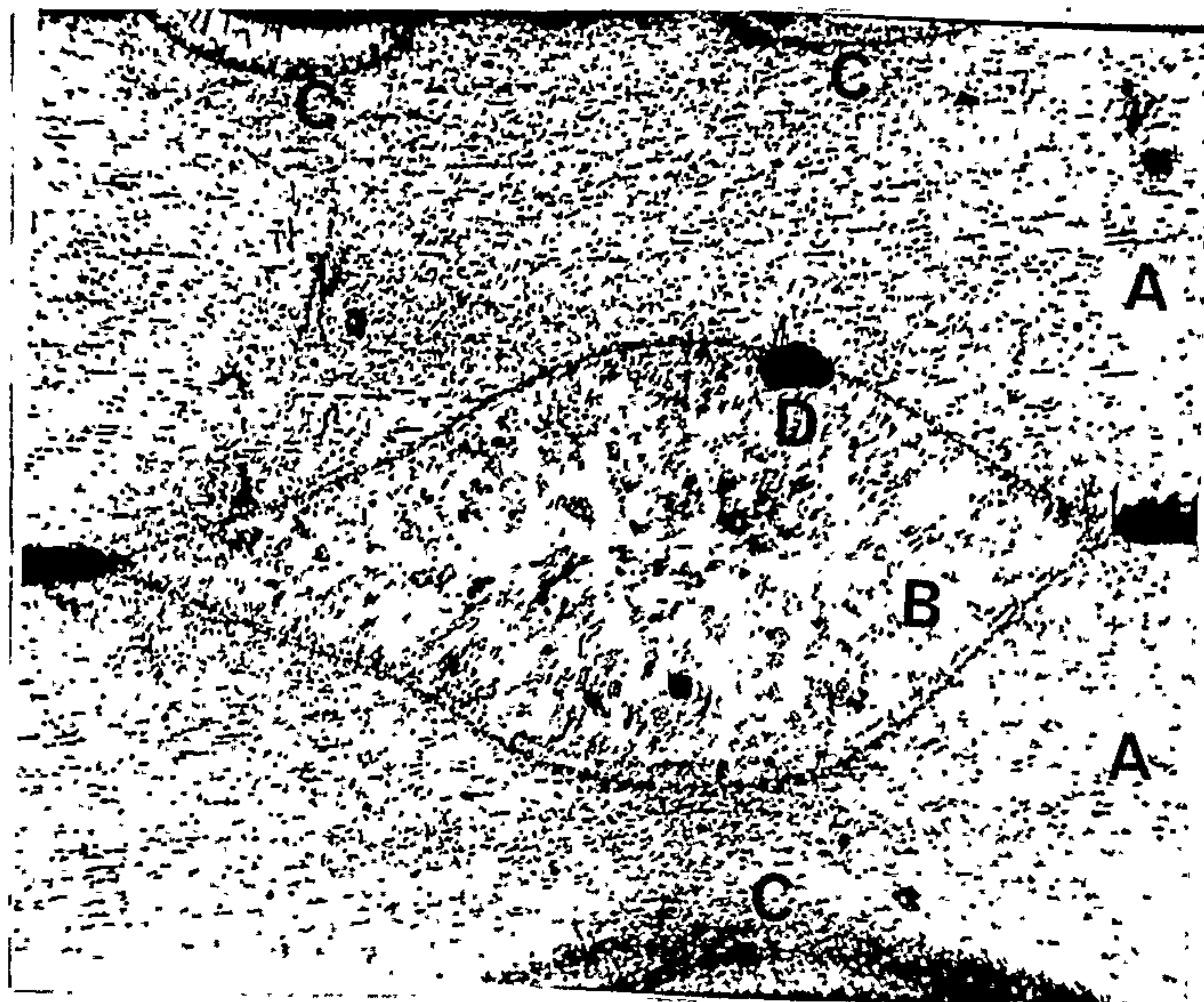


Figure 75.

Resistance welded stainless steel interface X 150.

Unitek 1050 Welder. Setting 7.

Legend.

- A. Unitek resilient flat wire
- B. Weld nugget
- C. Areas where burning occurred at surface of wire
- D. Porosity.

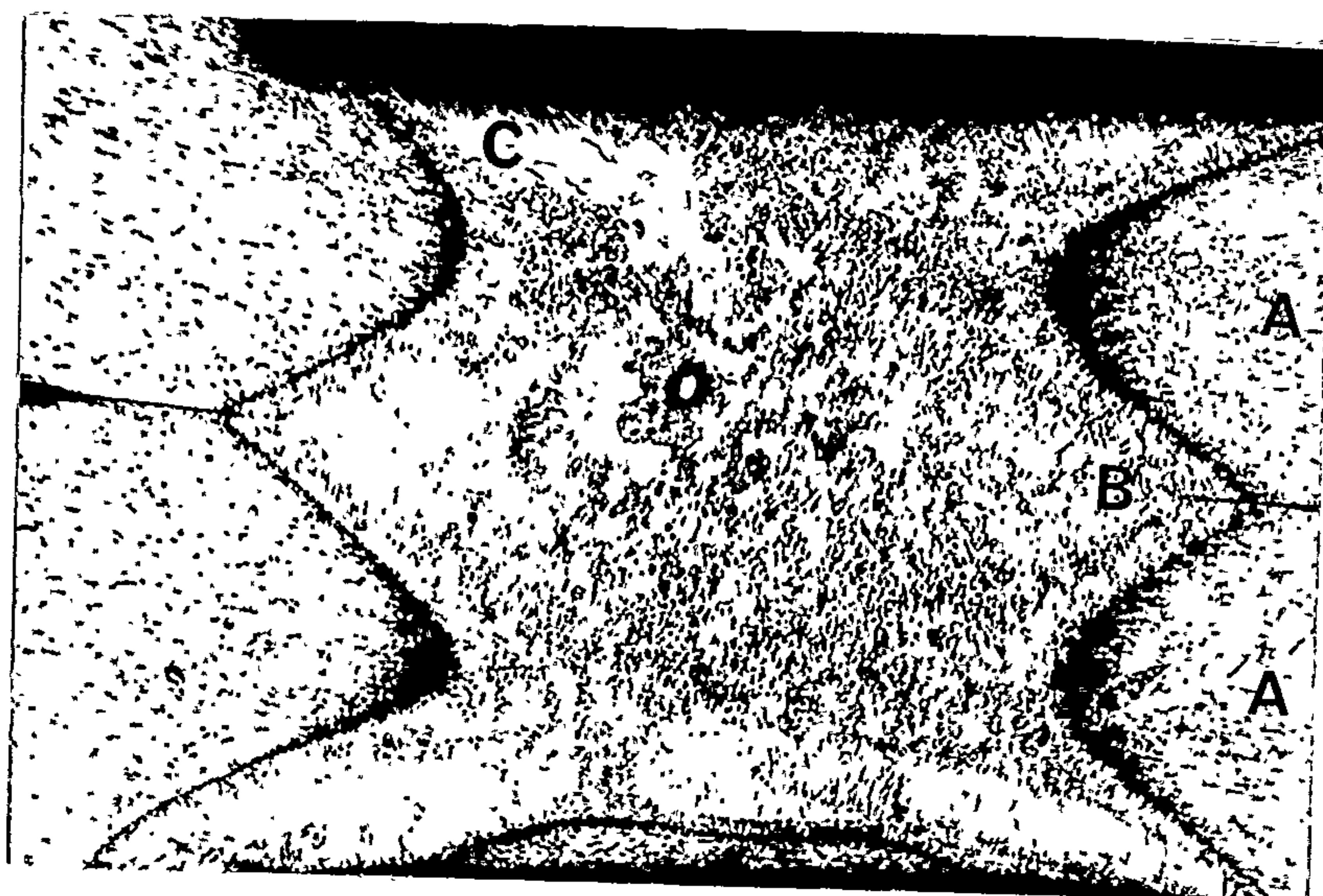
Figure 76.

Resistance welded stainless steel interface X 150.

Unitek 1050 Welder. Setting 9.

Legend.

- A. Unitek resilient flat wire
- B. Weld nugget
- C. Surface indentation.



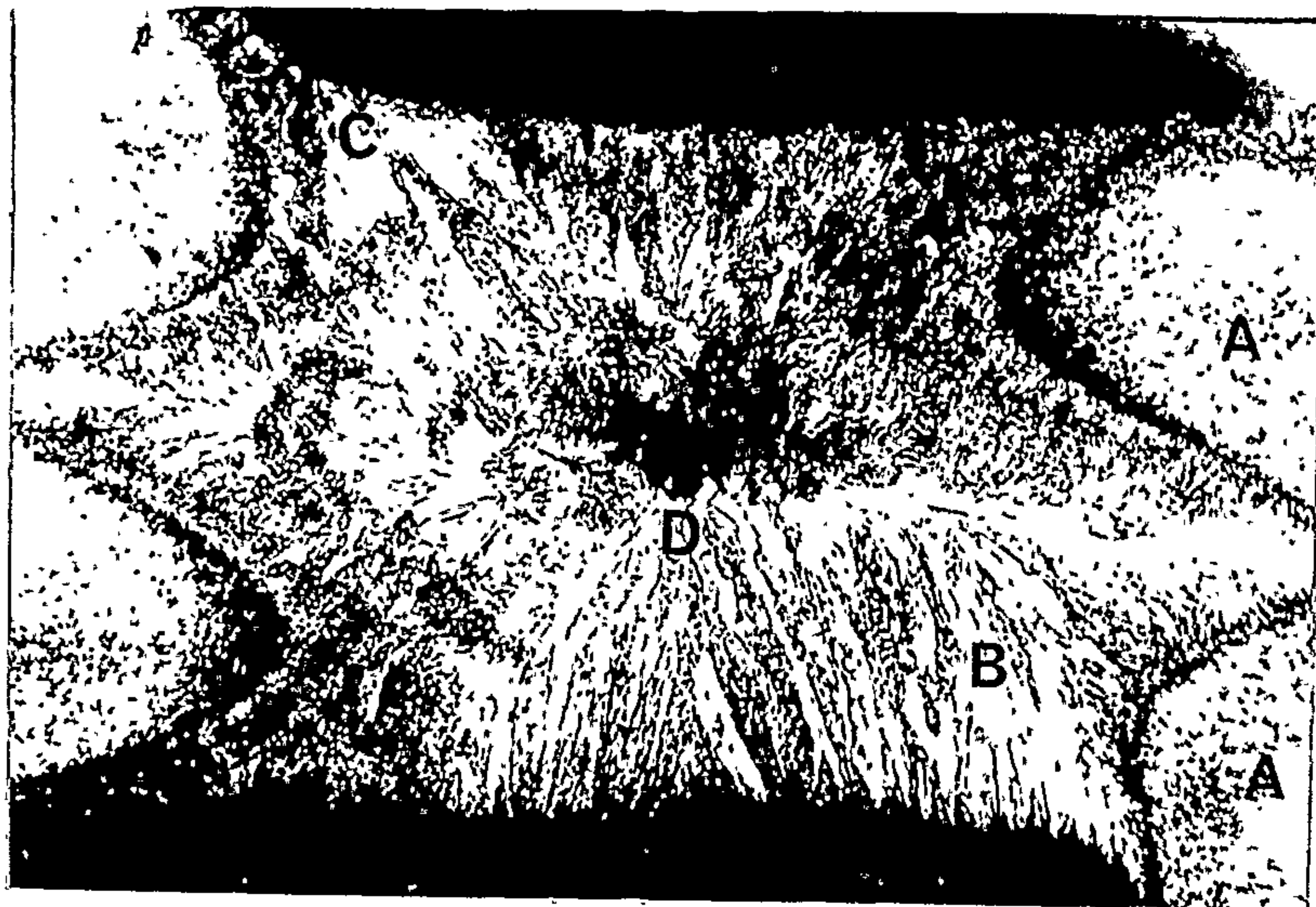


Figure 77.

Resistance welded stainless steel interface X 150.

Unitek 1050 Welder. Setting 10.

Legend.

- A. Unitek resilient flat wire
- B. Weld nugget
- C. Surface indentation
- D. Porosity.

Figure 78.

Resistance welded stainless steel interface X 300.

Rocky Mountain 650 Welder.

Heat selector 3 units (medium), 2 pulsations.

Legend.

- A. Rocky Mountain band material.
- B. Rocky Mountain bracket.
- C. Weld nugget showing two fusion areas.



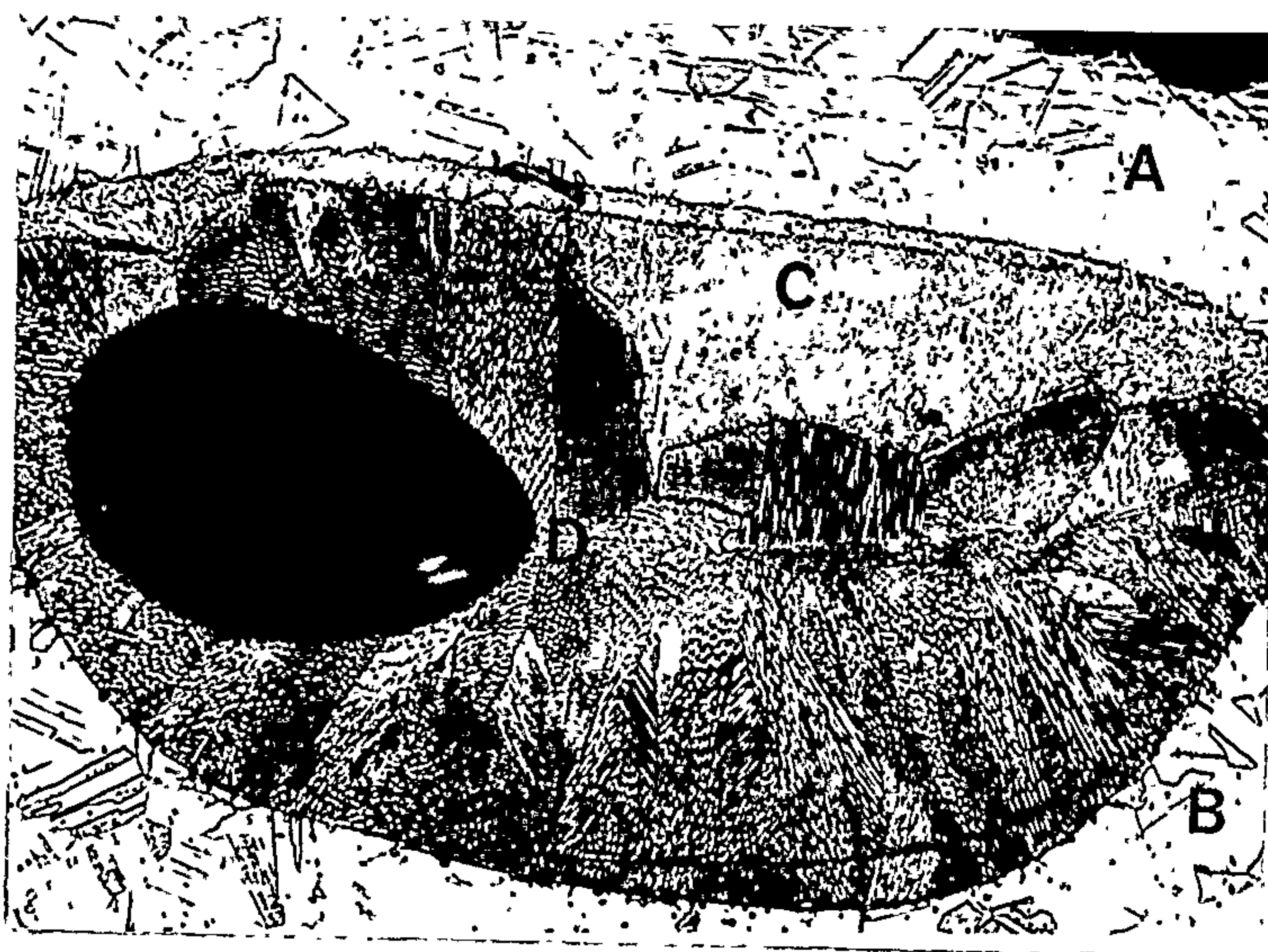


Figure 79.

Resistance welded stainless steel interface X 300.

Rocky Mountain 650 Welder.

Heat selector 3 units (medium), 6 pulsations.

Legend.

- A. Rocky Mountain band material
- B. Rocky Mountain bracket
- C. Weld nugget showing several fusion areas
- D. Porosity.

Figure 80.

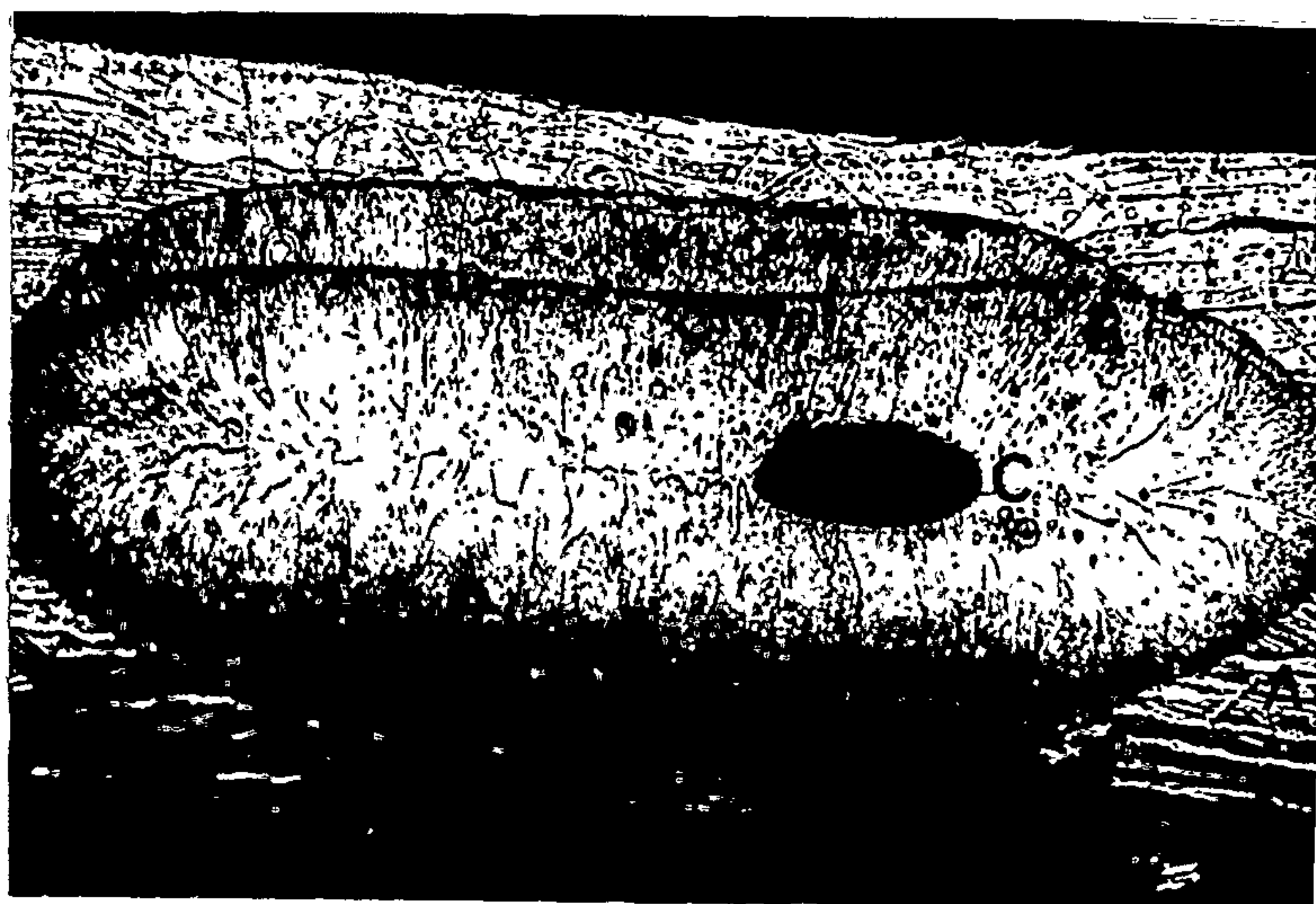
Resistance welded stainless steel interface X 300.

Rocky Mountain 650 Welder.

Heat selector 4 units (heavy), 4 pulsations.

Legend.

- A. Rocky Mountain band material
- B. Multi nugget formation
- C. Porosity.



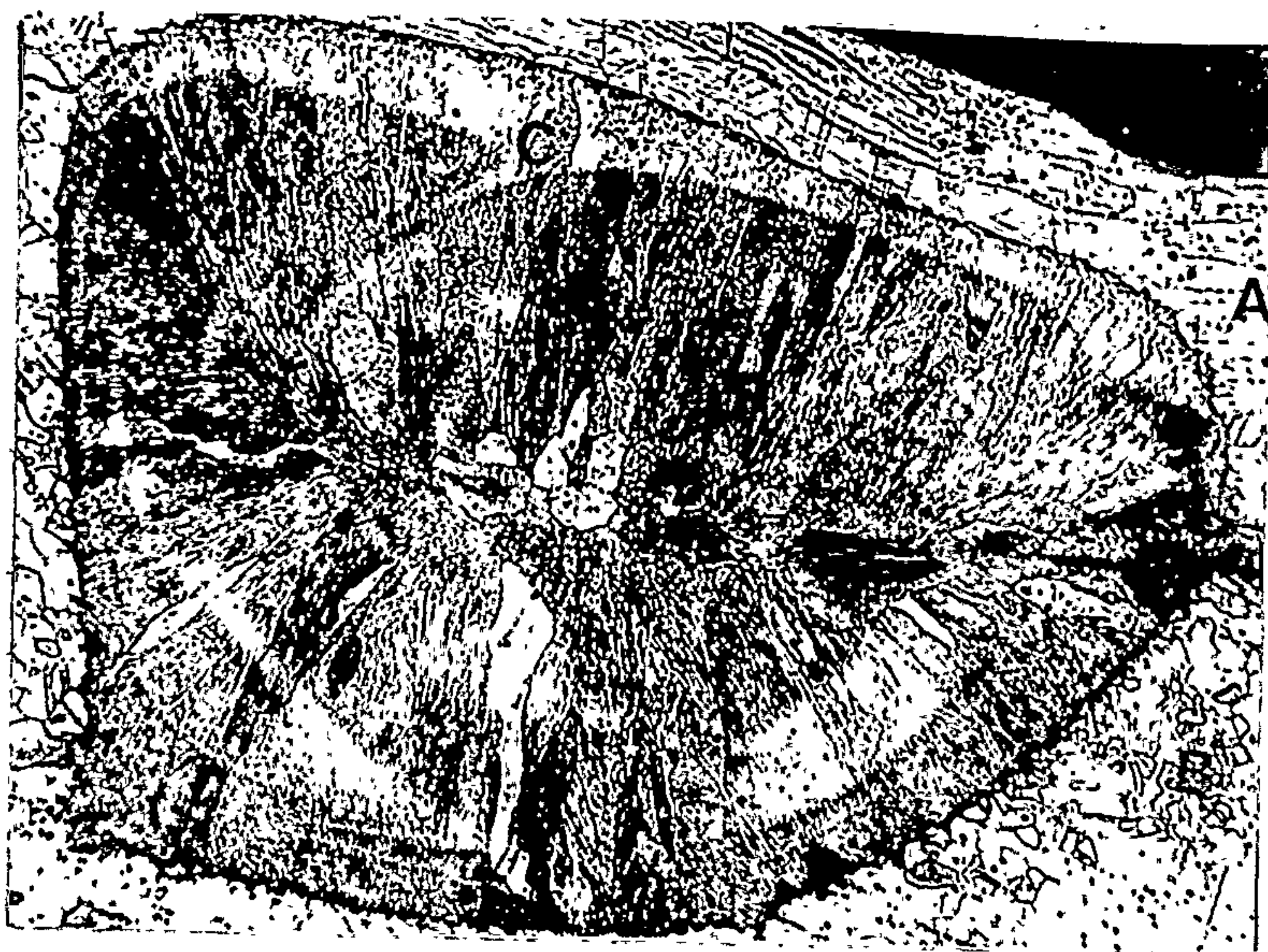


Figure 81.

Resistance welded stainless steel interface X 300.

Rocky Mountain 650 Welder.

Heat selector 4 units (heavy), 4 pulsations.

Legend.

A. Rocky Mountain band material

B. Rocky Mountain bracket

C. Multi-nugget formation.

Figure 82.

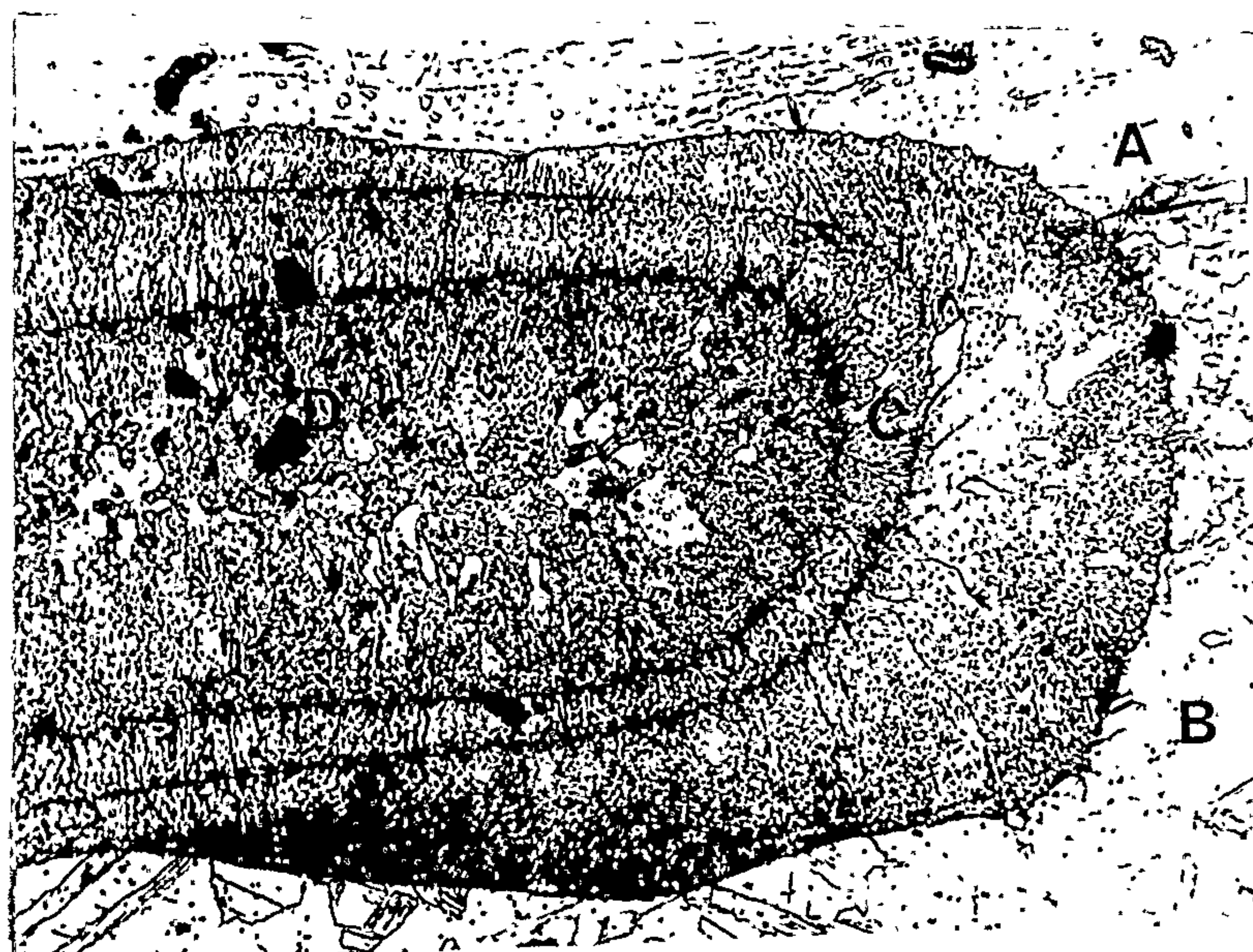
Resistance welded stainless steel interface X 300.

Rocky Mountain 650 Welder.

Heat selector 4 units (heavy), 6 pulsations.

Legend.

- A. Rocky Mountain band material
- B. Rocky Mountain bracket
- C. Multi nugget formation
- D. Porosity.



CONSIDERATION OF RESULTS

PART II. CHROMIUM CONTAINING ALLOYS.

CONSIDERATION OF RESULTS

PART II.

Chromium containing alloys.

In considering the types of bonding possible between metals Bien and Ayers⁵⁶ suggested the following classification.

Class I Autogeneous soldering

Class II Soldering by solution

Class III Soldering by wetting

a. absorption

b. diffusion

c. leaching

Class IV Electrochemical deposition

Nutting and Barker¹³⁴ state that if two pure metal surfaces, i.e. surfaces that are uncontaminated by oxides or any other combined or absorbed gases,¹³⁵ could be placed in perfect atomic contact, then they would be joined by the atomic bonds between them.

This is achieved by most methods used for joining metals. When brazing or soldering, a flux removes the surface contaminants and the liquid metal conforms to the interface of the parent metal.

Similarly, during electrodeposition, the deposited metal is in intimate contact with the basis metal.

Inter alloying is not necessary to form a strong bond between metals and it has been noted that "diffusion" and penetration of solder into the grain boundaries of the parent metal is undesirable.^{23,30}

Jackson¹³⁶ states that bond strength must be attributed to intimate contact between the two metals - the contact must be close enough to permit the interaction of the atomic forces between the atoms of the basis metal and the solder. "Provided that the bond is complete it does not matter whether it is one of atomic adhesion or solid solubility because the strength will not be less than that of the brazing alloy." However, the presence of a weak or brittle intermetallic film will adversely affect such a bond.

It has been observed by Vosmick and Taylor,⁴³ Henns,⁵² and Gardiner and Aamodt⁶¹ that in a silver soldered stainless steel joint the strength of the joint is in excess of the strength of the solder used. This led to the assumption that interalloying had occurred.

Cox and Setapen⁴⁸ considered the question of how a silver brazing alloy with an ordinary tensile strength of about 70,000 lbf./in² could withstand a stress of 130,000 lbf./in² and suggested the following theory:

"When brazing alloy is stressed it tends to elongate. However elongation requires a collateral reduction in area, and this reduction in area is restrained in the joint by the closely spaced hard faces of the steel to which the brazing alloy is bonded. The brazing alloy is then under triaxial tension. It has been established that a material under triaxial tension has a higher breaking strength along the principal axis of stress and a lower elongation than when pulled in simple tension. When the yield point of the steel is reached the steel elongates and collaterally reduces in cross section.

This then means a reduction of the lateral or radial tension stresses in the joint and the brazing alloy may then extend out to failure."

Milner¹³⁷ considers that this factor of "constraint" must be related to the tensile strength of brazed joints.

Case⁵¹ found that metallographic examination of conventionally made silver soldered joints failed to show measurable diffusion of the filler into the base metal. Longer cycles of up to four hours at 649°C gave clear indications of diffusion. By plotting the depth of penetration of the diffusion zone against the time of the heating cycle used he estimated that a one minute contact time caused an approximate diffusion depth of 4×10^{-5} inches (1.02μ).

Presuming these calculations to be correct the depth of penetration would be much less with dental appliances. Heating is discontinued as soon as the solder flows to minimise damage to the physical properties of the stainless steel.

The use of an electron microprobe analyser showed no evidence of diffusion within the 2 - 3 μ resolution of the probe. These results confirmed those of Laird and Fraunhofer.⁶⁴

Manko¹³⁸ considers that soldering below temperatures of 315°C depends upon wetting for the bond formation and requires neither diffusion nor the melting of the base metal to achieve bonding. He states that this is a reversible process and it is possible to open joints and separate the components by the application of heat. At temperatures above 426°C (brazing) the joining process relies upon wetting as well

as diffusion for the bond formation.

Skinner and Phillips¹⁴⁰ evaluating the literature conclude it is possible "that an atomic diffusion occurs from the wire to the solder during soldering, as well as from the solder to the wire. The situation is similar to that described for the gold soldered joints between gold alloy wires." The American Society for Metals¹⁴¹ state that "bonding results from an intimate contact produced by the dissolution of a small amount of base metal in the molten filler metal without fusion of the base metal."

The "notching" effect observed in this study at the grain boundaries of the stainless steel interface when gold or silver soldered indicates either an etching effect due to the flux used and, or, diffusion of the solder, when molten, into the grain boundaries of the stainless steel. Nucleation of both the gold and silver solder was influenced by the surface of the stainless steel.

Further investigation utilising the metallographic techniques described, variable heat saturation procedures and tapered sections could provide additional information.

Hothersall,^{141,142} Blum and Hogaboom,¹³² Gray¹⁴³ and Graham¹⁴⁴ have shown that epitaxy can also occur with electrodeposited metals when a well defined grain structure is present on the basis metal surface and equilibrium is established during the deposition of the coating.

During rapid deposition of metals, epitaxy does not occur, as the metal is laid down so fast that its grains must be nucleated rapidly.

Vegramyan and Petrova¹⁴⁵ state that provided no undue internal stresses are produced in the deposit, adhesion still exists.

Thus a relationship exists between the soldering and electroplating of the chrome containing alloys as a strong bond is formed between the parent and applied metal without any microscopical evidence of inter-alloying or diffusion.

Resistance welded stainless steel interface.

According to Skinner and Phillips,¹⁴⁶ Anderson,¹⁴⁷ and Vassar¹⁴⁸ the disadvantage of spot welding is the effect of the heat produced on the properties of the metal. Partial recrystallisation of the parent metal surrounding the nugget occurs in all welds due to dissipation of heat from the nugget. Further, these alterations in the structure of the stainless steel lead to tarnishing and corrosion and weld decay may occur.

Wilkinson¹⁴⁹ found that spot welding of high tensile stainless steel wire produced a joint only half as strong as a well-soldered joint because the wire was unduly softened with consequent loss of strength. Autogenous welding depends upon a localised union of the materials being joined through fusion or recrystallisation across the interface.

Phillips¹⁵⁰ considers that the minimum depth of penetration should not be less than 20% of the thickness of the materials being joined. This must also have a detrimental influence on the physical properties of high tensile materials.

In contrast, when welding a bracket to an orthodontic band, or welding bands where the primary consideration is attachment, a satisfactory and permanent union can be produced by spot welding.

In the specimens examined internal defects such as porosity were located within the nugget and therefore should not influence the strength of the weld.

An excessive concavity, pitting or burning of the surfaces in apposition to the electrodes, as seen in figures 74 and 75, indicates the use of excessive current. Apart from destruction of the physical properties of the stainless steel, excessive surface indentation causes a loss of weld strength due to reduction of the metal thickness in the weld area.

The grain structure of the nugget resembled that of a casting, presenting an equiaxed grain structure. Where a multi-pulse technique was used there was evidence of several overlaying nuggets.

SUMMARY AND CONCLUSIONS.

PART II. CHROMIUM CONTAINING ALLOYS.

SUMMARY AND CONCLUSIONS .

PART 11.

Chromium containing alloys .

1. Techniques have been developed and described for the examination of the interface between dissimilar metals, utilising electrolytic etching procedures.
2. Several interfaces have been examined that have not been recorded by other dental researchers.
i.e. a. gold solder - cobalt-chromium interface.
b. electroplated gold - cobalt-chromium interface.
c. electroplated gold - stainless steel interface.
3. A method of examining the interface between silver solder and stainless steel that permitted etching of both of the constituents of the soldered joint without loss of levelling has also been developed and described.
4. The grain boundaries at the surface of the stainless steel interface influenced the nucleation of the silver solder. Whilst no evidence of interalloying was visible, it is possible that diffusion did occur.
5. Surface activation of cobalt-chromium and stainless steel was essential to produce a satisfactory bond with electrodeposited gold.
6. Anodic etching of cobalt-chromium alloy during electroplating produced an irregular surface and the electroplated gold closely followed the conformation of the cobalt-chromium to provide an additional keying effect.

7. The same anodic etching procedure when applied to stainless steel similarly produced an irregular surface but the electroplated gold did not follow the conformation of the stainless steel interface. A possible conclusion for this phenomenon is that in the case of stainless steel a microporous layer was present at the interface, due to preferential dissolution of the iron, chromium and nickel content. During the etching procedure stainless steel shows less resistance to chemical attack than the cobalt-chromium alloy.
8. This variation in surface passivation between stainless steel and cobalt-chromium was also evident with the gold soldered specimens. A tighter union occurred between gold soldered cobalt-chromium than with gold soldered stainless steel. In the latter case the gold solder did not follow the surface irregularities present on the stainless steel. The flux used for soldering produced a similar effect as the anodic etching procedure.
9. The metal continuity established by spot welding differed from the other interfaces investigated as the union was formed by fusion whilst the parts being joined were firmly held together. An autogenous nugget of fused parent metal formed the joint and if multiple pulses were used more than one nugget was produced. The metallographic structure of the nugget differed from that of the wrought parent metal and was characterised by the equiaxed grain structure of a casting.

MATERIALS AND METHODS

PART III. CREVICE CORROSION OF SILVER
SOLDERED STAINLESS STEEL.

MATERIALS AND METHODS.

PART III. Crevice corrosion of silver soldered stainless steel.

Fifty soldered joints for each of the following series were prepared for crevice corrosion testing.

- (a) Silver soldered stainless steel.
- (b) Silver soldered, nickel plated stainless steel.
- (c) Silver soldered, gold plated stainless steel.
- (d) Silver soldered stainless steel, gold plated after soldering.
- (e) Silver soldered, gold plated stainless steel replated with gold after soldering.

A cold drawn austenitic stainless steel^{*} was used and the specimens that required electroplating prior to silver soldering (b, c, d, and e) were prepared by the technique described by Rogers⁹¹ for the gold plating of cobalt-chromium alloys.

The silver soldered "T" joints were prepared using the technique described in Part I.b. Group d was ultrasonically cleaned after soldering and then surface activated, nickel plated, and gold plated. Group e did not require further surface activation after silver soldering as the only exposed surfaces were gold and silver solder - the soldered joint was cleaned ultrasonically to remove any traces of flux and then gold plated.

* Remanit Dentarum. 19 gauge.

Flux.

Comweld Silver Brazing Flux No. 2 was used as in Part II.a.

Silver Solder.

The silver solder used was Easy Flow No. 2 - the same as in Part II.c.

The composition requirements for silver solder (A.D.S. T30-1964) are:

	Minimum %	Maximum %
Silver.	40	80
Copper.	10	35
Zinc.	10	30
Cadmium.	-	20
Nickel.	-	3.0
Total other elements.	-	0.5

It will be noted that the solder used does not conform to the above standard due to its high cadmium content. This will be discussed later.

Accelerated corrosion test.

After preparation the joints were inspected, cleaned ultrasonically in non-ammoniated detergent, washed and dried. The technique as described by Bien and Ayers⁵⁶ was used for corrosion testing. The specimens were placed in a solution having the following formula:

Anhydrous ferric sulphate.	8g.
Hydrofluoric acid.	4ml.
Distilled water.	96ml.

After 24 hours they were removed, rinsed, dried and inspected for separation.

Microscopical examination.

The mounting and polishing of the specimens was the same as previously described in Part I.b. After washing and drying the specimens were etched by using the Struers Relapol and Struers RP2 electrolyte for 2-3 minutes at a current density of 0.4 amp./dm^2 .

They were then cleaned with distilled water, dried and examined microscopically.

Determination of electropotential between silver solder and surfaces involved in soldering.

A calculated length of stainless steel wire and silver solder, presenting an area of one square inch (5.25 cm^2), was placed in 200ml. of the accelerated corrosion testing solution and the electropotential between the two metals was measured with a sub-standard millivoltmeter.

This was repeated and the electropotential between nickel plated stainless steel, silver solder and gold plated stainless steel, silver solder was determined.

RESULTS.

PART III. CREVICE CORROSION OF SILVER SOLDERED STAINLESS STEEL.

RESULTS

PART 111. Crevice corrosion of silver soldered stainless steel.

Results of the experiments have been shown in tables I to IV and figures 83 to 91. The silver soldered, stainless steel interface and the electro-deposited gold stainless steel interface have been previously examined and discussed in Part 11.c. and Part 11.e.

TABLE I. Accelerated corrosion test after 24 hours.

Group	Treatment of Stainless Steel	Satisfactory		Unsatisfactory	
		No.	%	No.	%
a.	Silver soldered	9	41	18	82
b.	Nickel plated then silver soldered	13	37	26	74
c.	Nickel and gold plated, and silver soldered	42	8	84	16
d.	Silver soldered and nickel and gold plated	33	17	66	34
e.	Nickel plated, gold plated silver soldered and gold plated	50	Nil	100	Nil

TABLE II shows the number of successful specimens taken for microscopic examination after the 24 hour test. The remaining specimens were kept for eighteen months in a controlled atmosphere of 21°C and a relative humidity of 55-65%. It was noted that after this period of time further specimens had failed.

Group.	a	b	c	d	e
No. of specimens taken from Table I.	3	4	6	6	3
Remaining specimens.	6	9	36	27	47
No. that failed after 18 months.	6	5	18	Nil.	Nil.
Percentage failure rate	100	55.6	50	Nil.	Nil.

The combined total failure rate of both the 24 hour test and the specimens after the eighteen month period is shown in table III.

TABLE III.

Summary of failure of soldered joints.

Group.	a	b	c	d	e
Failed 24 hours	41	37	8	17	Nil.
Failed 18 months	6	5	18	Nil.	Nil.
Expected failure of specimens taken for microscopic exam.	3	2	3	Nil.	Nil.
Total failures	50	44	29	17	Nil.
% Failures	100	88	58	34	Nil.

The electropotential between silver solder and the surfaces presented on the stainless steel when placed in the accelerated corrosion testing solution is shown in table IV.

TABLE IV. Electropotential between silver solder and surfaces used in soldering procedures. The Electrolyte was the solution used for accelerated corrosion testing.

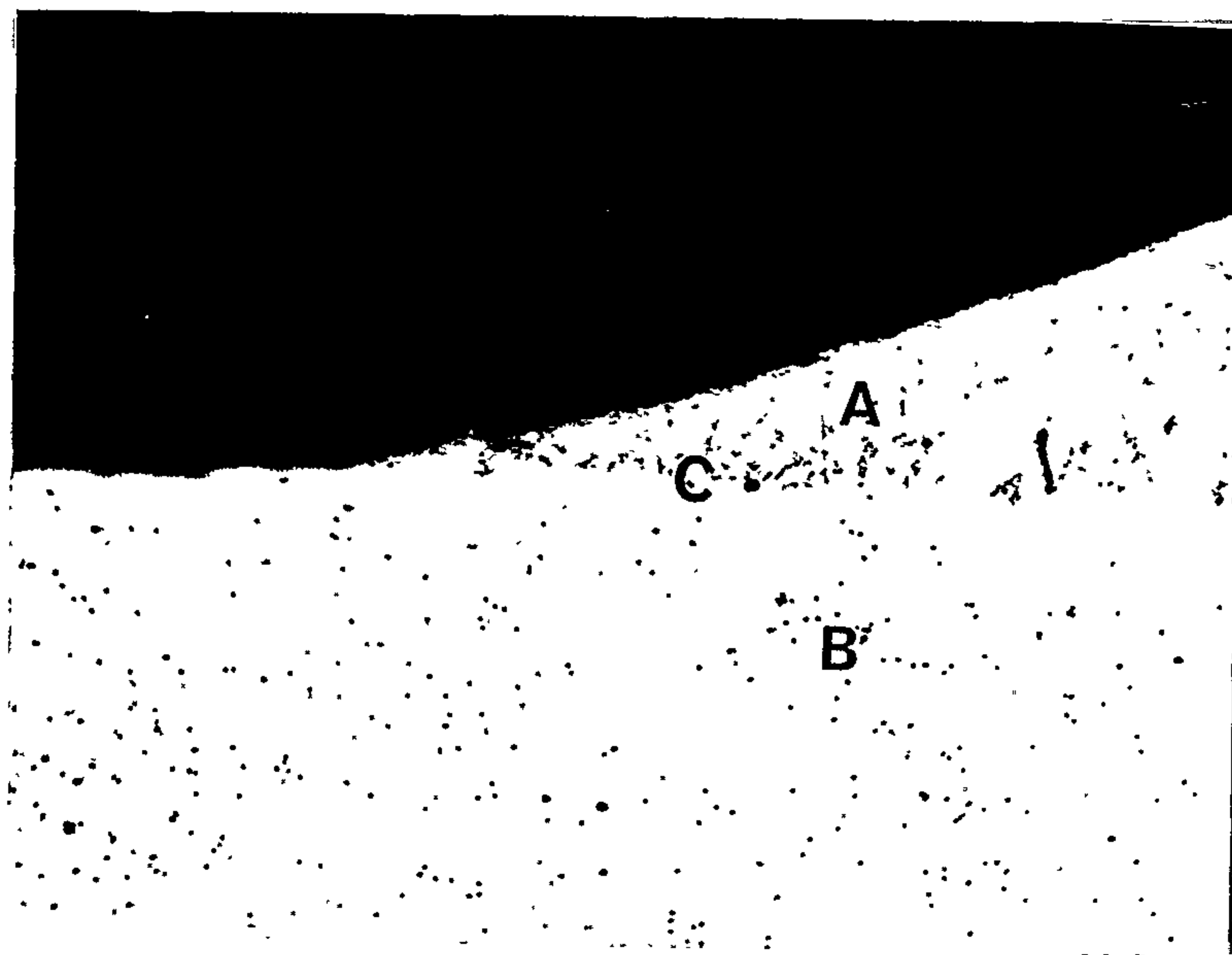
Surface presented to silver solder	Reading in millivolts	Anode
Stainless steel	64	stainless steel
Nickel	26	silver solder
Gold	242	gold

Figure 83.

A tight silver solder, stainless steel joint X 100.

Legend.

- A. Silver solder
- B. Stainless steel
- C. Interface.



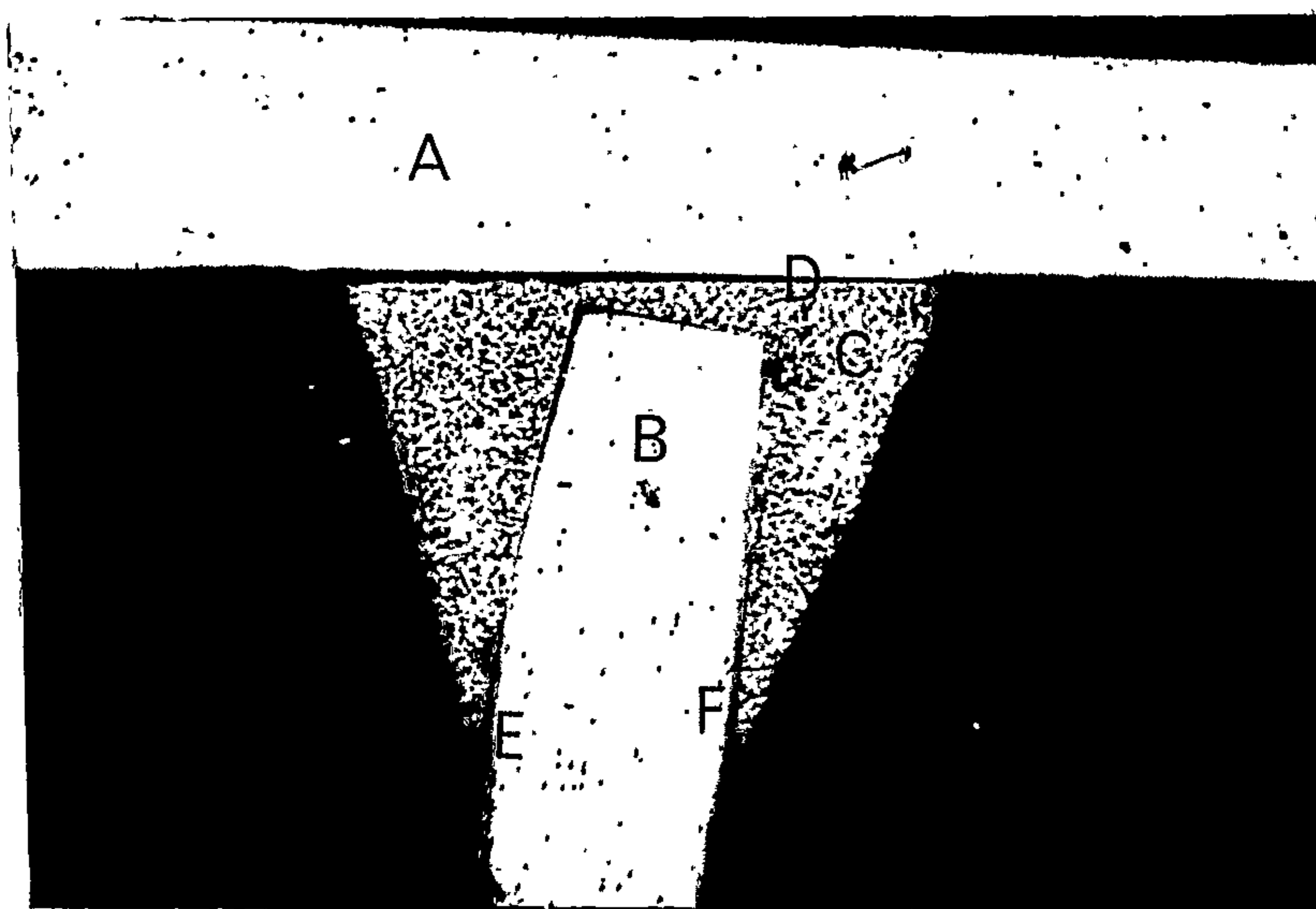


Figure 84.

Crevice corrosion between silver solder and stainless steel X 20.

Legend.

A. and B. Stainless steel.

C. Silver solder

D. Interface - complete separation

E. Interface - crevice corrosion

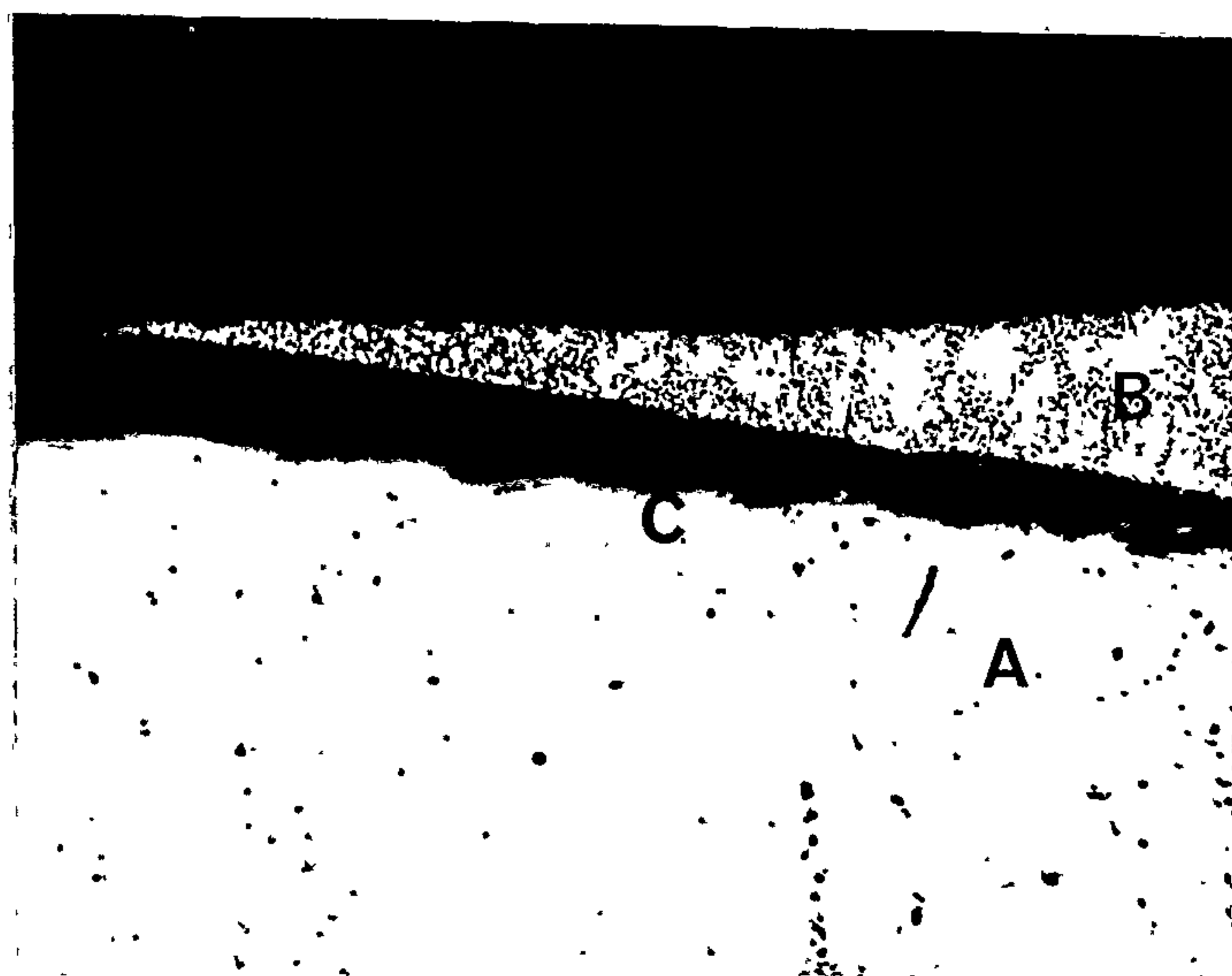
F. Interface - crevice corrosion

Figure 85.

Interface E of figure 82 X 200.

Legend.

- A. Stainless steel
- B. Silver solder
- C. Interface shows irregularity of the stainless steel surface.



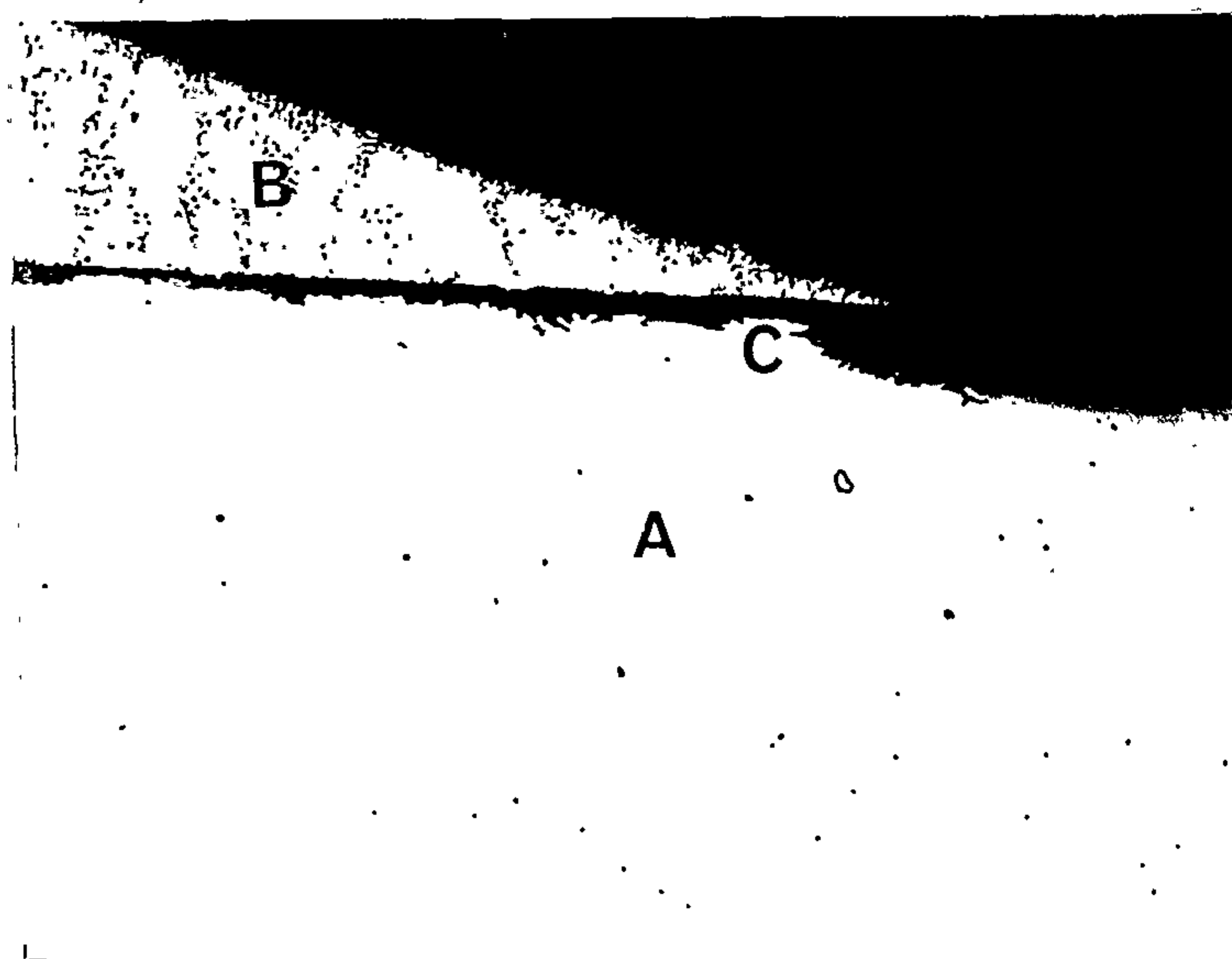


Figure 86.

Interface F of figure 82 X 200.

Legend.

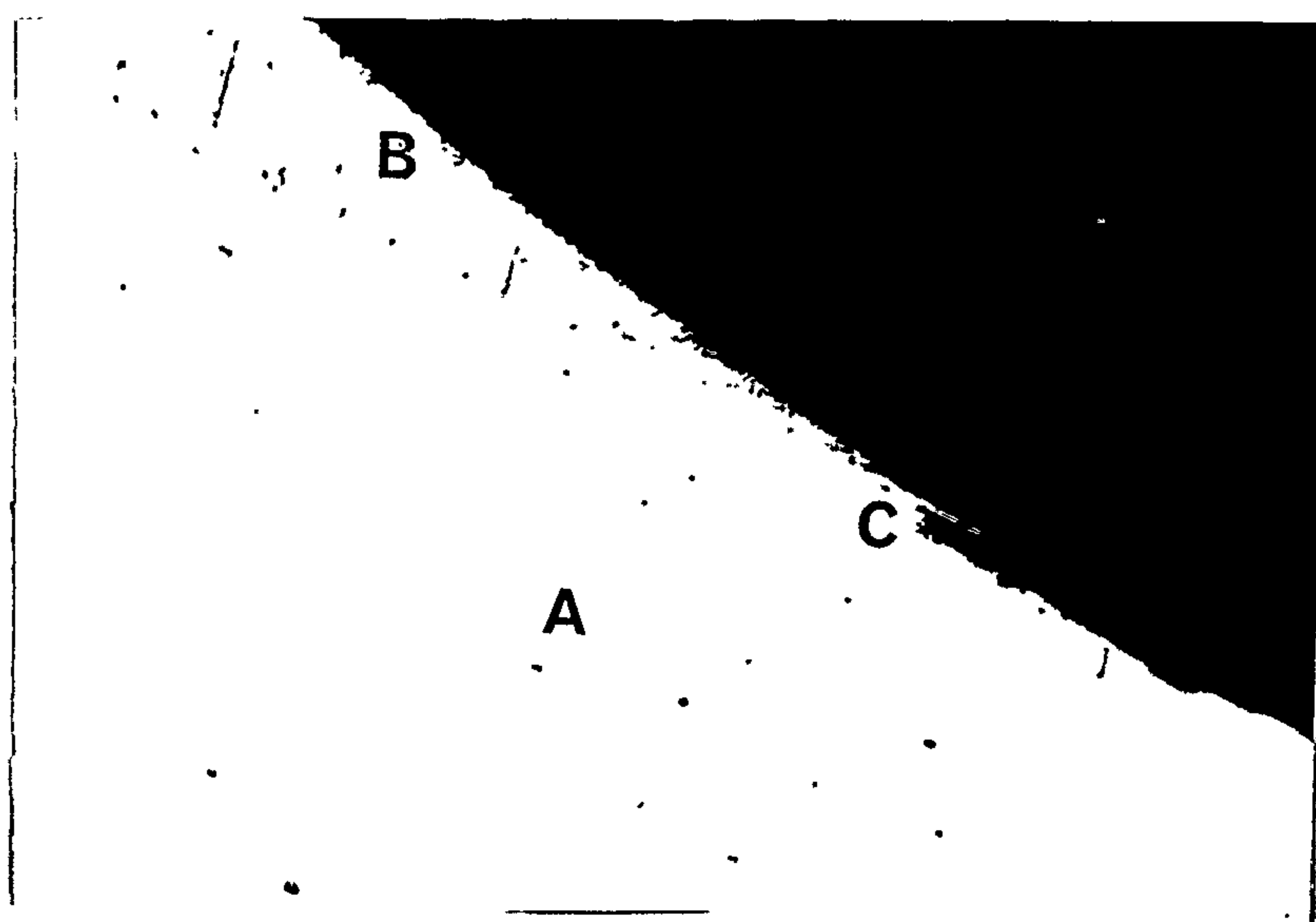
- A. Stainless steel
- B. Silver solder
- C. Dished area in the stainless steel.

Figure 87.

Silver soldered stainless steel X 100.

Legend.

- A. Stainless steel
- B. Silver solder
- C. Dished area in the stainless steel with a thin spine of silver solder above it. The attack has been initiated in the stainless steel and the silver solder has not been affected.



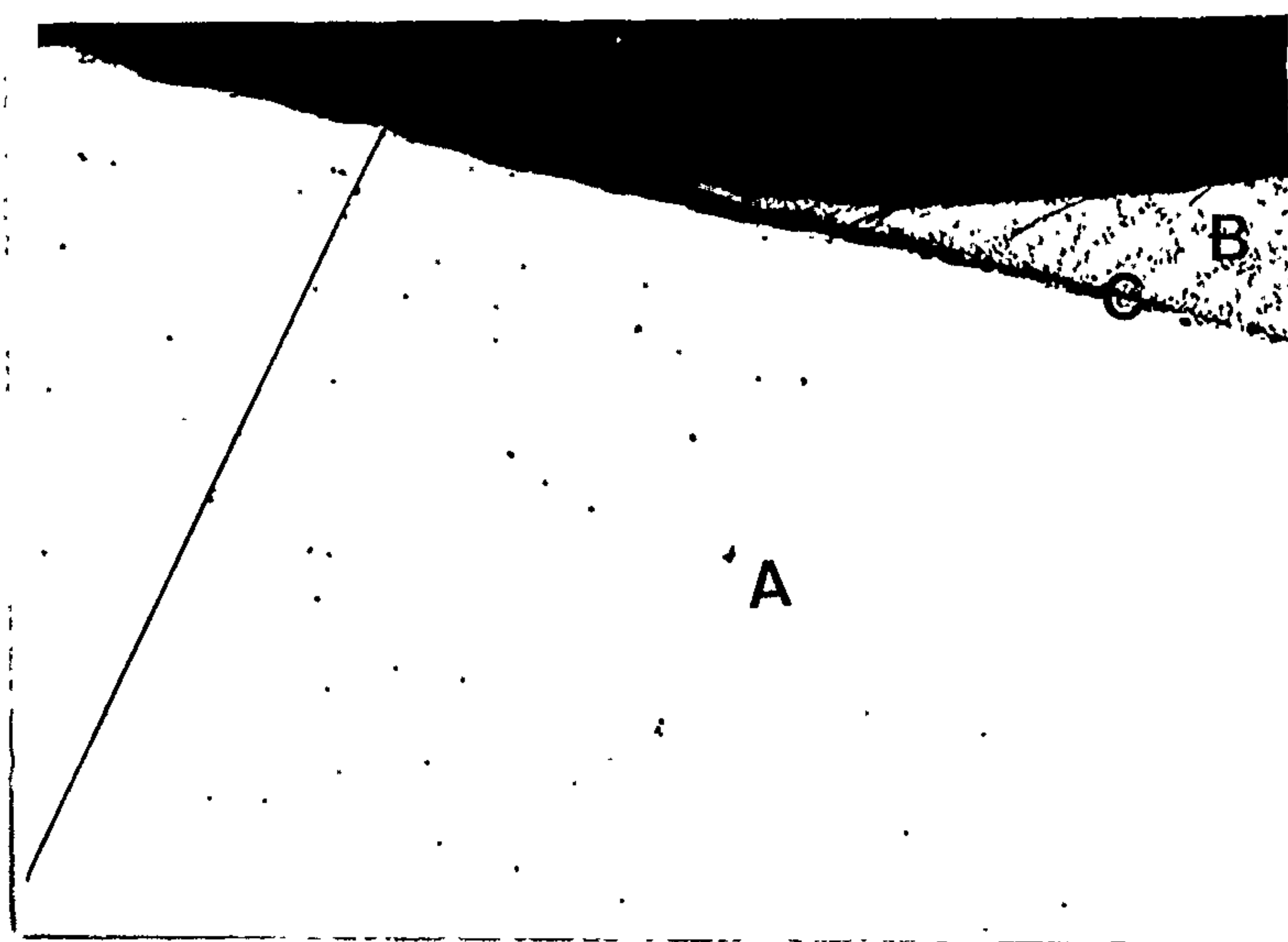


Figure 88.

Silver soldered, nickel plated stainless steel X 100.

Legend.

- A. Stainless steel
- B. Silver solder
- C. Crevice corrosion.

Figure 89.

Stainless steel, gold plated prior to silver soldering X 100.

Legend.

- A. Stainless steel
- B. Silver solder
- C. Electrodeposited gold
- D. Continuity between the gold and the silver solder.

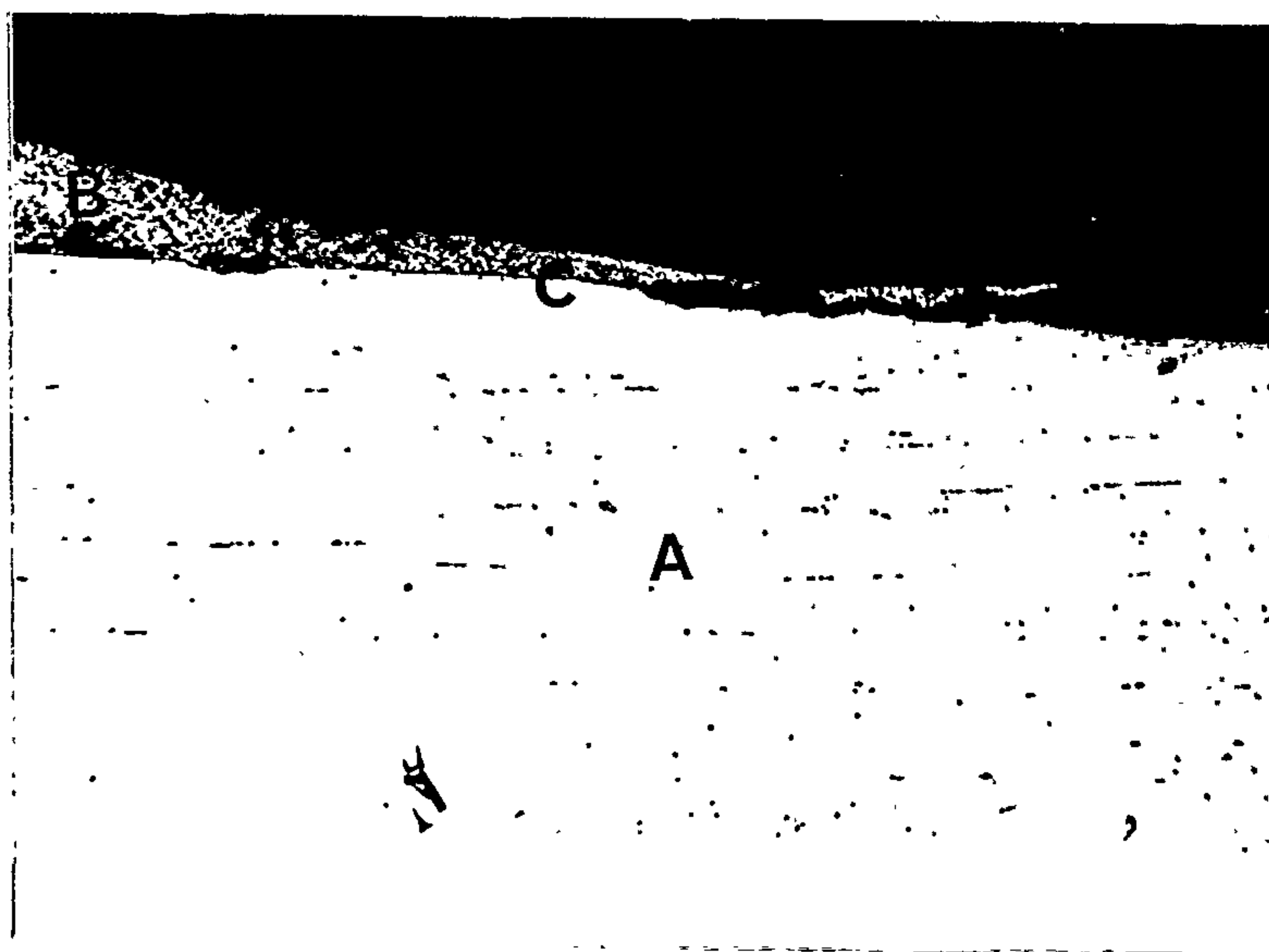


Figure 90A.

Gold plated after silver soldering the stainless steel x 100.

Legend.

- A. Stainless steel
- B. Silver solder
- C. Crevice corrosion - both interfaces show signs of chemical attack.



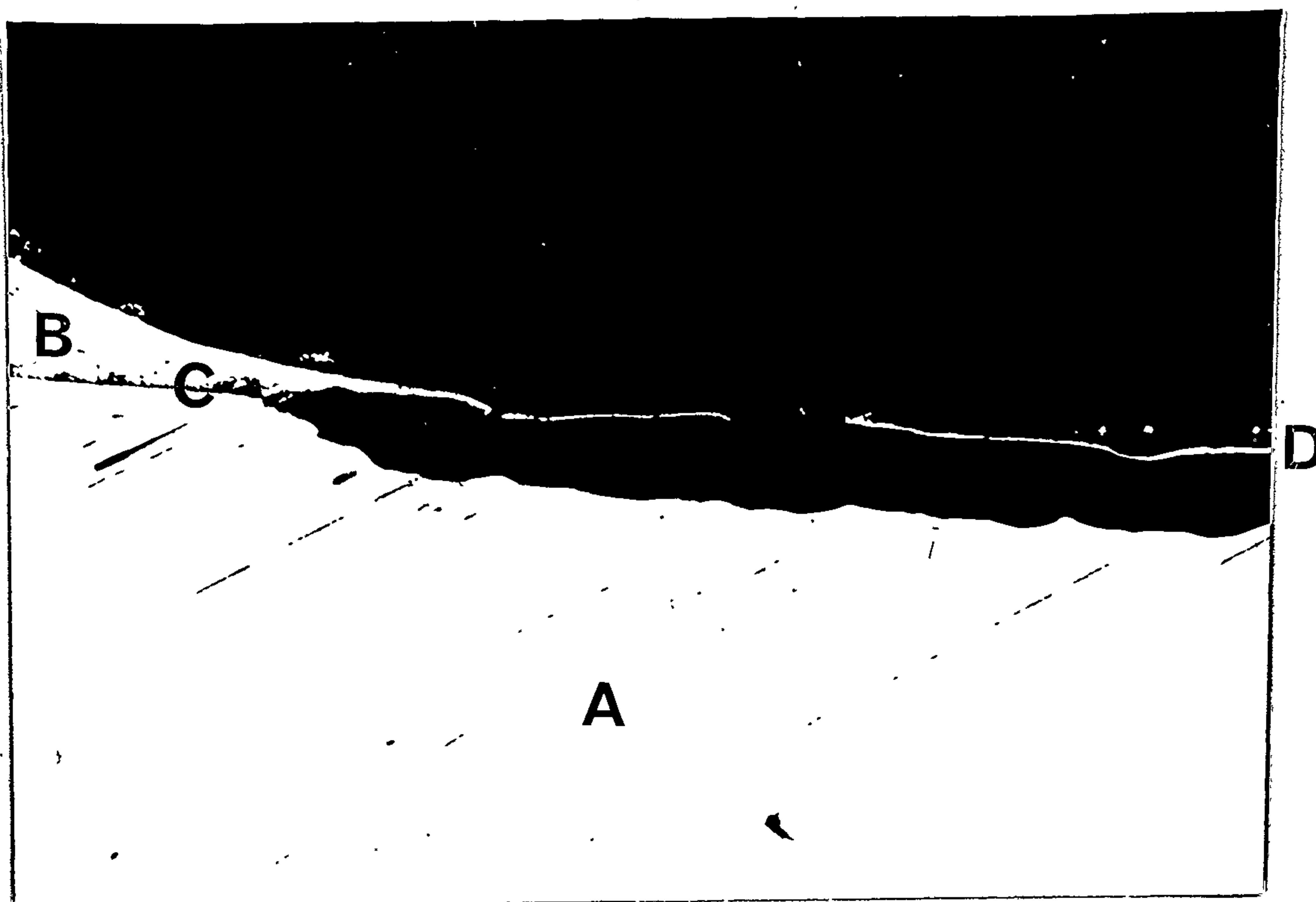


Figure 90B.

Control specimen (x 100) gold plated after silver soldering the stainless steel, but not subjected to the accelerated corrosion test.

Legend.

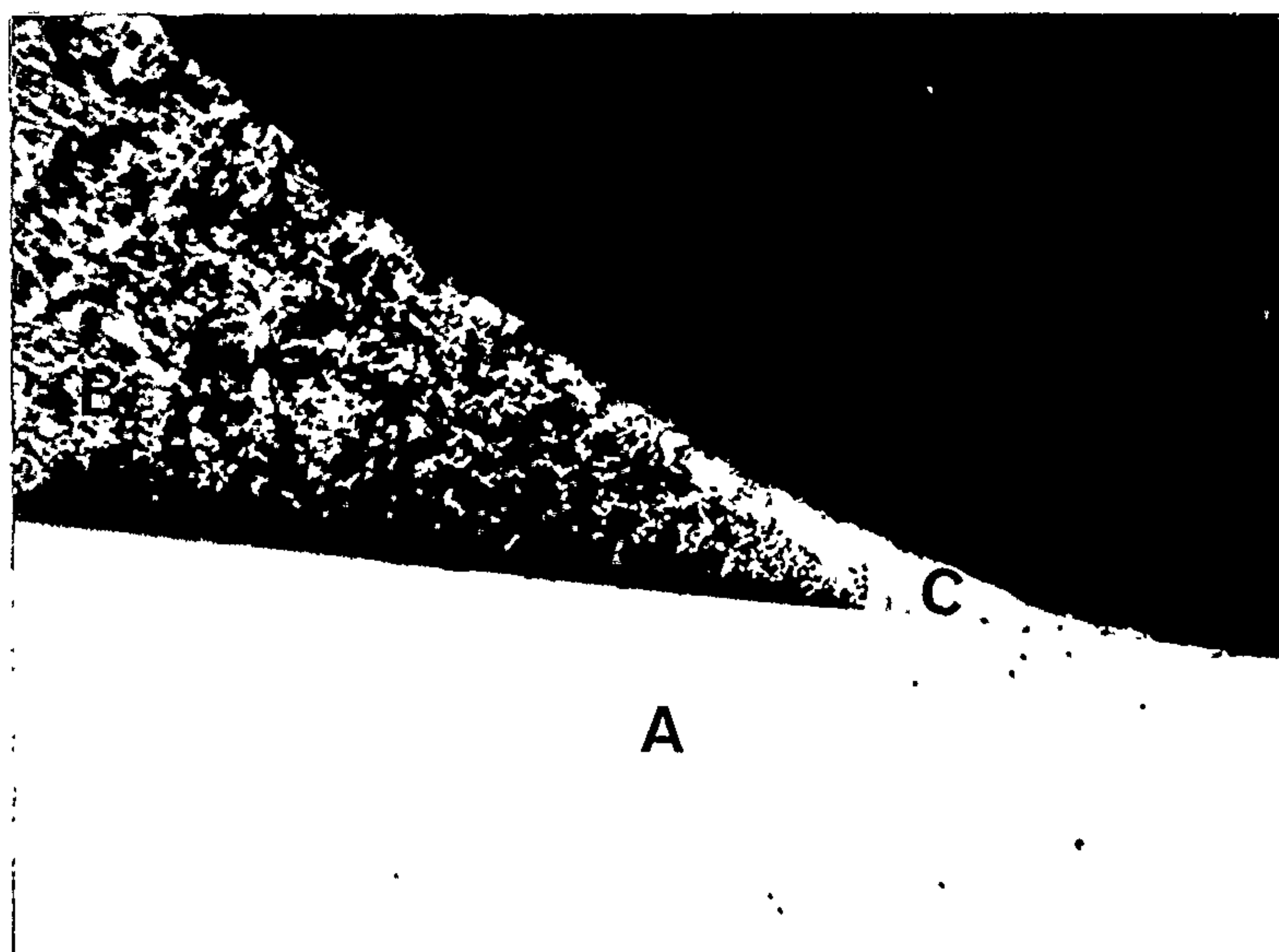
- A. Stainless steel
- B. Silver solder
- C. Crevice corrosion - both interfaces show signs of chemical attack.
- D. Electrodeposited gold.

Figure 91.

Gold plated stainless steel that has been silver soldered and then replated. X 100.

Legend.

- A. Stainless steel
- B. Silver solder (heavily etched)
- C. Electroplated gold.



Group (a) shows the extreme results of crevice corrosion with an 82% failure after the 24 hour test and complete failure of all the specimens after an eighteen month interval.

Figure 83 shows a tight joint between the silver solder (a) and the stainless steel (b). There is no evidence of crevice corrosion.

Figure 84 shows several stages of crevice corrosion. Complete separation has occurred between the stainless steel (a) and the silver solder (c) at the interface (d). Early stages of crevice corrosion are shown at the junction of the silver solder (c) and the stainless steel (b) at the interface (e) and (f).

Figure 85 is a higher magnification of the interface (e) in figure 84. The interface between the stainless steel (a) and the silver solder (b) shows marked irregularity of the stainless steel, whilst the silver solder is virtually unaffected except for the degree of separation from the stainless steel.

Figure 86 is a higher magnification of the interface (f) in figure 84. The stainless steel interface (a) has the same notched appearance but the dished area (c) shows greater dissolution of the stainless steel at the margin of the silver solder (b). The silver solder interface shows no irregularity.

Figure 87 shows an early phase of crevice corrosion between the stainless steel (a) and silver solder (b). As in figure 86 a dished out area (c) is evident and the stainless steel is showing preferential dissolution to the silver solder.

CONSIDERATION OF RESULTS.

PART III. CREVICE CORROSION OF SILVER
SOLDERED STAINLESS STEEL.

Group (b) does indicate some advantage due to the presence of nickel and, or, the uniformly activated surface of the stainless steel. Figure 88 shows crevice corrosion (c) when nickel plated stainless steel (a) has been silver soldered (b). The stainless steel does not show evidence of irregular dissolution as seen in the previous figures. The nickel layer is not visible at this magnification.

Group (c) shows that the gold plating of stainless steel prior to silver soldering does not control crevice corrosion and the failure rate increased from 16% to 58% over the eighteen month interval. In figure 89 the stainless steel (a) has a smooth surface. The gold layer (c) has continuity at the margin of the silver solder (d) and the structure of the silver solder (b) shows interalloying.

Group (d) was gold plated after silver soldering the stainless steel and shows more resistance to crevice corrosion after the eighteen months period. Figure 90A shows a marked similarity to the type of corrosion present in group (a). The stainless steel (a) has been attacked leaving an irregular interface (c) and this specimen also shows dissolution of the silver solder interface (b). Crevice corrosion in group (d) was caused by anodic activation in the Woods' nickel electrolyte prior to gold plating. To verify this conclusion ten additional specimens were prepared but they were not subjected to the accelerated corrosion test. Identical results were obtained as shown by the specimen in figure 90B.

Group (e) shows control of crevice corrosion. The final layer of gold protected the joint during the accelerated corrosion test. Figure 91 shows stainless steel (a) silver solder (b) and the protective layer of electrodeposited gold (c). The silver solder has been heavily etched to show the gold.

CONSIDERATION OF RESULTS.

PART III. Crevice corrosion of silver soldered stainless steel.

Crevice corrosion is initiated by electrochemical action at the interface between the dissimilar metals of the joint. It is difficult to assess the role of the flux used in soldering, but the presence of flux or its by-products would enhance and localise this corrosive action. After the crevice is formed the corrosion of the solder joint could be accelerated by an oxygen cell type of differential aeration but this would be superimposed upon the existing condition and could not be considered an initiating factor.

When stainless steel is silver soldered without prior surface treatment and crevice corrosion occurs, the stainless steel beneath the solder has an irregular, notched appearance and the breakdown of the joint is due to a preferential dissolution of the stainless steel. (Figures 85, 86, 87, and 90). This is further shown in Table IV where the stainless steel is anodic to the silver solder when placed in the accelerated corrosion testing electrolyte. In figures 86 and 87 the stainless steel is dished at the silver solder margin, but in figures 85 and 90 there is no preferential dissolution in this area.

In figure 88 where the surface of the stainless steel was activated and a thin protective nickel coating applied prior to soldering, the stainless steel shows no sign of interfacial attack. The silver solder has become anodic and corrosion is due to ionic dissolution of the

silver solder.

Table IV confirms that the silver solder is anodic to the nickel plated stainless steel. The presence of nickel in Tables I.c. and III.c. shows a slight decrease in the failure rate. Bien and Ayers⁵⁶ used a silver solder containing 2% nickel (Lynco solder - Ag 40%, Cu 30%, Zn 28% and Ni 2%. Melting range 661-769⁰C) and their failure rate was in the order of 20%. The high flow point of this solder would preclude its use for modern dental soldering procedure, and this will be discussed later. Figure 90 shows dissolution of both silver solder and the stainless steel interfaces. The entire joint was made anodic during the surface activation procedure by passing a current of 3.2 amp./dm². for two minutes in the Woods' electrolyte. This caused both alloys to be electrolytically attacked. The accelerated corrosion test was ineffective due to the continuous gold surface and the crevice corrosion was caused by the activating procedure. Consequently there was no change in the failure rate, when re-examined after the eighteen months period.

Thus three variations of electrochemical attack causing crevice corrosion have been observed. During accelerated corrosion testing of the silver solder - stainless steel joint (group a.) the stainless steel was anodic and corroded. When the stainless steel was nickel plated (group b.) the silver solder became anodic and the basis metal showed no signs of corrosion. Both the silver solder and the stainless steel (group d.) showed anodic dissolution during surface activation treatment prior to electroplating.

A low temperature soldering technique is essential with stainless steel to minimise the precipitation of carbides and to reduce the softening that occurs.^{60,151} Wilkinson⁵⁹ found a marked variation in the soldering temperatures recorded between operators and optimal results were at least 30°C above the flow point of the solder. The silver solder used for these experiments has a lower melting range than most solders available, but because of its high cadmium content toxicity must be considered. Gold plating, as in group e, apart from preventing crevice corrosion, eliminates the problem of toxicity as the solder is covered by a protective layer of gold. The use of gold plated stainless steel wire also assists the silver soldering procedure. The quality of a solder junction is influenced by the ability of the solder to flow into small spaces by capillary action. As silver solder readily alloys with gold, this ensures intimate contact with the basis metal. The development and use of modified solders is now possible because the final protective gold coating eliminates undesirable biological effects. Further the surface tarnishing and corrosion of the solder is prevented. Silver solders are available with a lower flow point, but they tend to have a wide melting range (LM5.* - Ag 63%, Cd 37%, melting range $340-400^{\circ}\text{C}$; LM15.* - Ag $33\frac{1}{3}\%$, Cd $33\frac{1}{3}\%$, Zn $33\frac{1}{3}\%$, melting range $280-320^{\circ}\text{C}$) and their tensile strength is respectively $\frac{1}{4}$ and $\frac{1}{3}$ that of the solder used in this experiment. The development of a more

* Matthey Garrett Pty. Ltd.

suitable silver solder does seem both desirable and possible.

If manufacturers supplied gold plated stainless steel orthodontic bands, wires and crown forms, the technique would be simplified to a gold plating operation after silver soldering.

Gold plated stainless steel bands and crowns will withstand manipulation and contouring if electroplated by the technique described but the final protective gold surface should consist of one of the harder electrodeposited golds^{88,89} for greater durability in the oral environment.

SUMMARY AND CONCLUSIONS.

PART III. CREVICE CORROSION OF SILVER
SOLDERED STAINLESS STEEL.

SUMMARY AND CONCLUSIONS

PART 111. Crevice corrosion of silver soldered stainless steel.

1. Various electroplating procedures have been investigated as a means of controlling crevice corrosion between silver solder, stainless steel joints.
2. The accelerated corrosion test described caused three variations in electrochemical attack upon the joint, and this was directly related to the electropotential of the surfaces presented to the electrolyte.
3. Crevice corrosion was prevented by electroplating with gold both before and after silver soldering the stainless steel.
4. In the technique described the stainless steel surface was activated and protected by the first layer of electrodeposited gold. The silver solder alloyed with this gold layer so a second application of electrodeposited gold was required to protect the joint from electrolytic action.
5. As the oral environment can also be protected from the silver solder by the electrodeposited gold, solders that would otherwise be biologically unacceptable can be used. The silver solder used in this experiment has a low melting range (608-617°C) but a high cadmium content. As tissue compatibility is assured the emphasis can now be placed on the physical properties of the solder to minimise softening of the stainless steel and precipitation of carbides.
6. Crevice corrosion and tarnishing of the soldered joint has been eliminated by the electroplating procedure described.

GENERAL CONCLUSIONS.

GENERAL CONCLUSIONS

The basic object of this investigation was to examine metallographically the nature of the interfaces formed in metallic unions that may have a dental application.

Throughout all of the metal interfaces examined, microscopically visible interalloying was not required to form an efficient joint. The bonding process was dependent upon wetting and diffusion occurring at the interface and a relationship was found to exist between the microscopic surface irregularities, such as grain boundaries, that occur on the surface of the parent metal, and the grain structure of the joining metal.

These surface irregularities were accentuated by the process of anodic etching or by the chemical action of the flux on the parent metal during the joining procedure.

Gross diffusion and interalloying was possible between the gold alloys but it is generally accepted that the resultant change in composition could be accompanied by a loss of physical properties.

The surface of the basis metal had a marked effect on the nucleation of the solders investigated. Epitaxy occurred with gold solder and the notched grain boundaries influenced the nucleation of the silver solder.

The metallographic investigation provided techniques for examining the interfaces between dissimilar metals.

In Part 1, an evaluation of the union between cast gold alloys and an electroformed gold matrix was carried out and a satisfactory casting

procedure was derived from the results.

The electrolytic polishing and etching procedures evolved will have a further application to the evaluation of porosities encountered when casting gold alloys and for the study of gold soldered unions.

In Part 11, a technique was developed specifically for the metallographic study of the stainless steel, silver solder interface. This method of using electroplated gold to protect the chemically active metal during the electrolytic polishing and etching procedures could be applied to many interfaces that have different degrees of chemical activity.

In Part 111, a metallographic examination on the occurrence of crevice corrosion between stainless steel and silver solder, permitted a greater understanding of its initiation and the development of a technique that eliminated this type of corrosion. This investigation utilising a protective electroplated gold surface over the soldered joint has a significant relevance to the biological aspects of dental treatment.

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PUBLICATIONS.

ELECTROFORMING A GOLD MATRIX FOR INDIRECT INLAYS

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ELECTROFORMING has been used in dentistry since 1840.¹ The technique has provided a durable die surface of either copper or silver created by electroforming the impression and making a die of the tooth.²⁻⁸ This report describes an electroplating process which forms a gold matrix that has a minimum thickness of 0.005 inch. The matrix is produced on a die of the prepared tooth, and it finally becomes an integral part of the completed casting (Figs. 1 and 2).

CLINICAL USE OF ELECTROFORMED GOLD MATRIX

The electroformed gold matrix makes possible a new and different indirect inlay technique. The dentist makes a rubber-base impression and pours a Densite or Hydrocal die of the tooth in the usual way. The entire surface of the cavity preparation and margins is then metalized by means of a silver paint[†] to provide a conducting surface⁹ on which gold is electrodeposited to the required thickness (0.005 to 0.01 inch). The gold matrix thus formed is returned to the prepared tooth in order to check the accuracy of the matrix and to burnish its margins. This is particularly advantageous in the gingival areas.

The wax pattern is completed within the electroformed gold matrix. A narrow margin of the soft gold matrix is left extending from the wax pattern, which may be safely returned to the tooth for a recheck. The inlay is then completed by casting a suitable gold alloy into the matrix (Figs. 3 and 4). The cast gold forms an intermediate alloy with the pure gold matrix.

For pinlay preparations, gold pins are inserted into the metalized die prior to electroforming.

ELECTRODEPOSITION OF GOLD

Although mainly used for decorative purposes, electrodeposited gold has found application in dentistry⁷ because of its resistance to chemical attack and for use where a pure gold color is desired on nonprecious metals or alloys of gold. Such deposits, which are usually produced from cyanide solutions of low gold concentration, are very thin—approximately 0.000001 to 0.00001 inch. The maximum current density is limited to 0.86 amp. per square decimeter (8 amp. per square foot), and the rate of deposition is low. Thick deposits from these solutions tend to be dull, require periodic burnishing, and are relatively soft.

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†Degussa conductive silver 200.

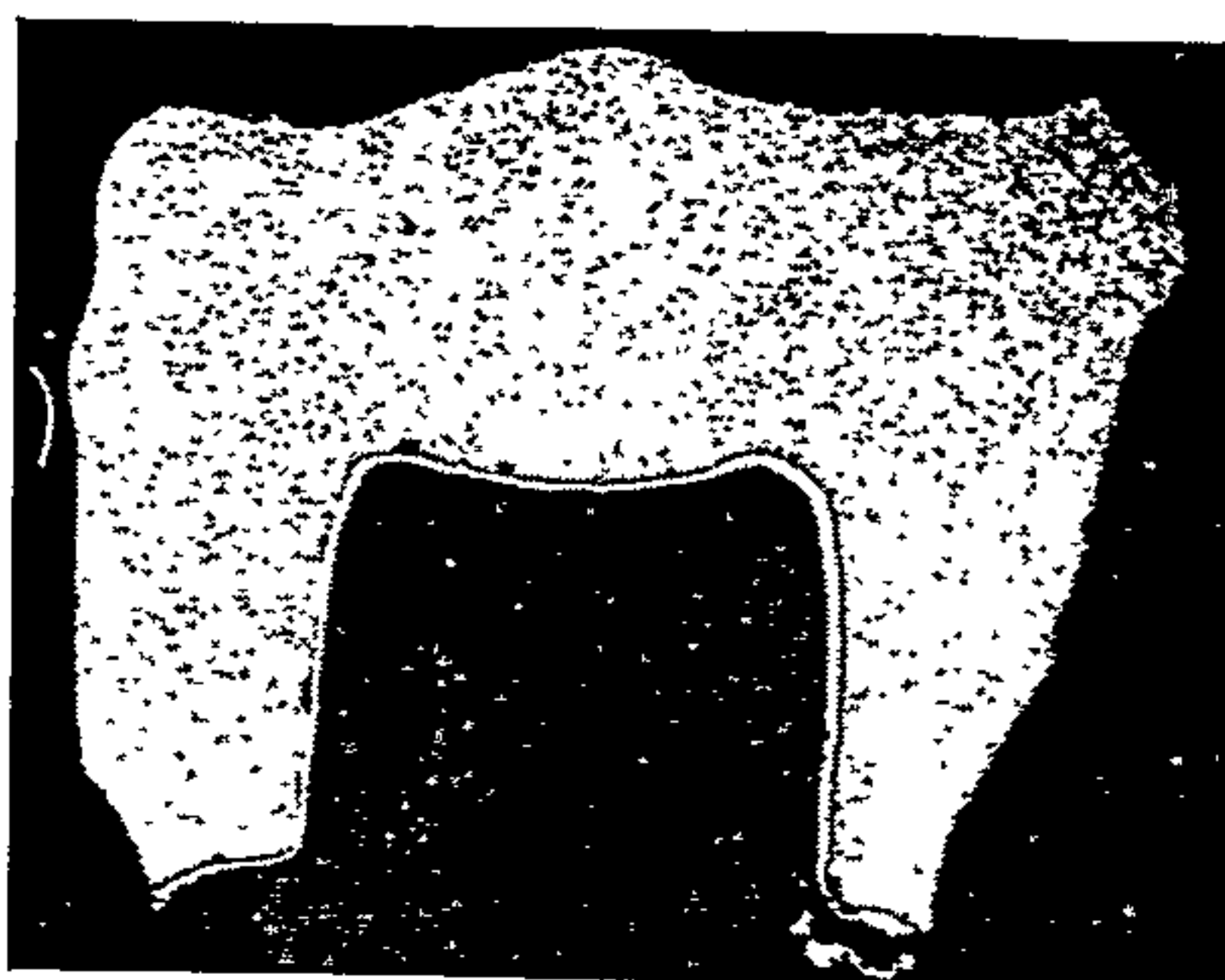


Fig. 1.—A section of a completed casting. The apparent gap between the gold matrix and cast alloy is due to differential etching. ($\times 10$; reduced $\frac{1}{3}$.)



Fig. 2.—A section showing the interfacing of the gold matrix and the cast alloy. ($\times 50$; reduced $\frac{1}{3}$.)

An important war development was the application of as much as 0.001 inch of gold on radar equipment.¹⁰ In recent years, much attention has been focused on the production of thick, bright deposits of pure gold¹¹ and gold alloys¹²⁻¹⁷ for use in the electronic and communication industries.

EQUIPMENT FOR FORMING THE GOLD MATRIX

The gold electroplating solution was contained in a 1 L. tank* immersed in a water bath which was electrically heated and thermostatically controlled. A mechanical stirrer provided rapid agitation. A rolled gold sheet was used as the anode. Direct current was supplied by a rectifier which was controlled by a variable transformer for experimental determinations, but a 2 V. lead storage battery and rheostat were used for producing the matrices (Figs. 5 and 6).

ELECTROPLATING SOLUTIONS

Several exploratory experiments were conducted with gold plating solutions. Gold chloride solutions containing 6.3 Gm. per liter (1 ounce per gallon) of

*Perspex.

gold and conventional cyanide solutions containing up to 3.15 Gm. per liter (0.5 ounce per gallon) of gold were used initially, but even at high temperatures, the current density was limited to 0.86 amp. per square decimeter. Current densities in excess of this produced granular and loosely adherent deposits. The production of smooth deposits from these solutions was slow, and the process required continuous attention.

The solution proposed by Kushner^{18,19} gave smooth deposits 0.003 inch thick when a current density of 2.15 amp. per square decimeter (20 amp. per square foot) was used at a temperature of 57° C. Higher current densities produced coarse, burned deposits. The deposits contained 1 to 3 per cent nickel and tended to be too brittle for dental use.

In view of these results and because the presence of nickel introduced an additional variable, the nickel salt was eliminated. The free cyanide concentration was further increased to permit the use of higher current densities. Satisfactory matrices were obtained from the solution shown in Table I.

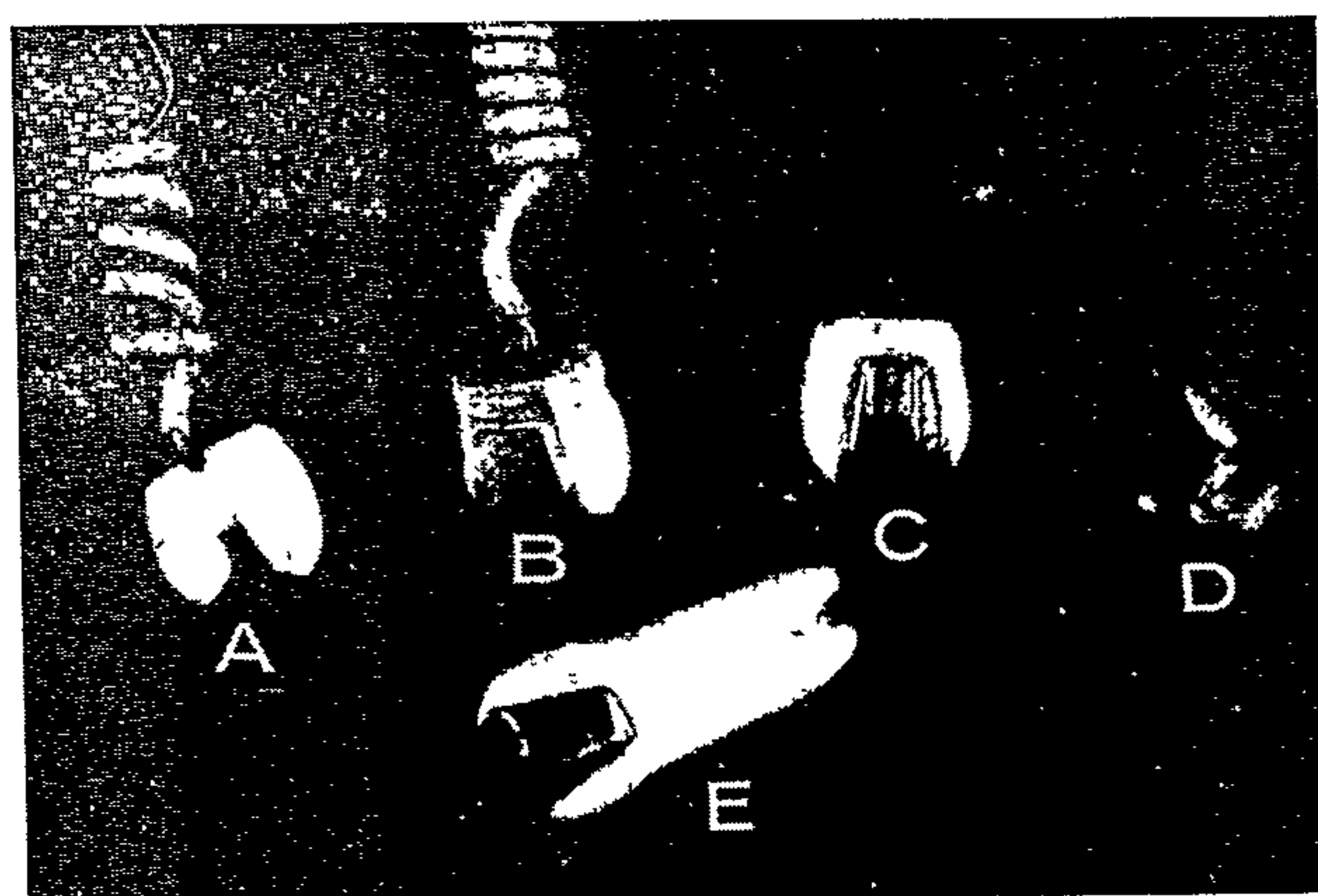


Fig. 3.—Steps in the inlay procedure: (A) a die with the conducting wire attached; (B) the metalized die; (C) the electroformed matrix on the die; (D) the wax pattern contoured and carved in the gold matrix; (E) the completed casting on the tooth.

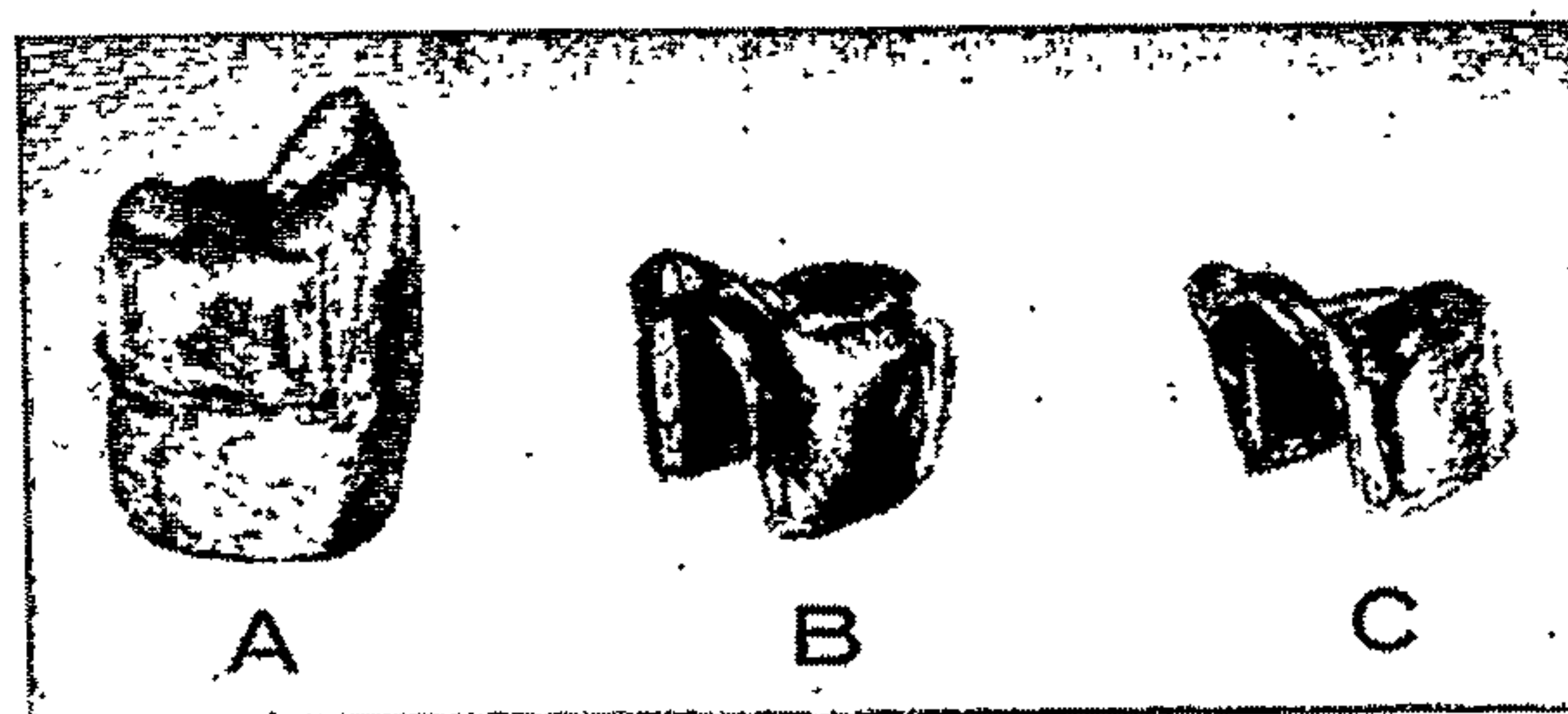


Fig. 4.—(A) The die of the prepared tooth; (B) the completed inlay cast on the matrix; (C) the gold matrix.

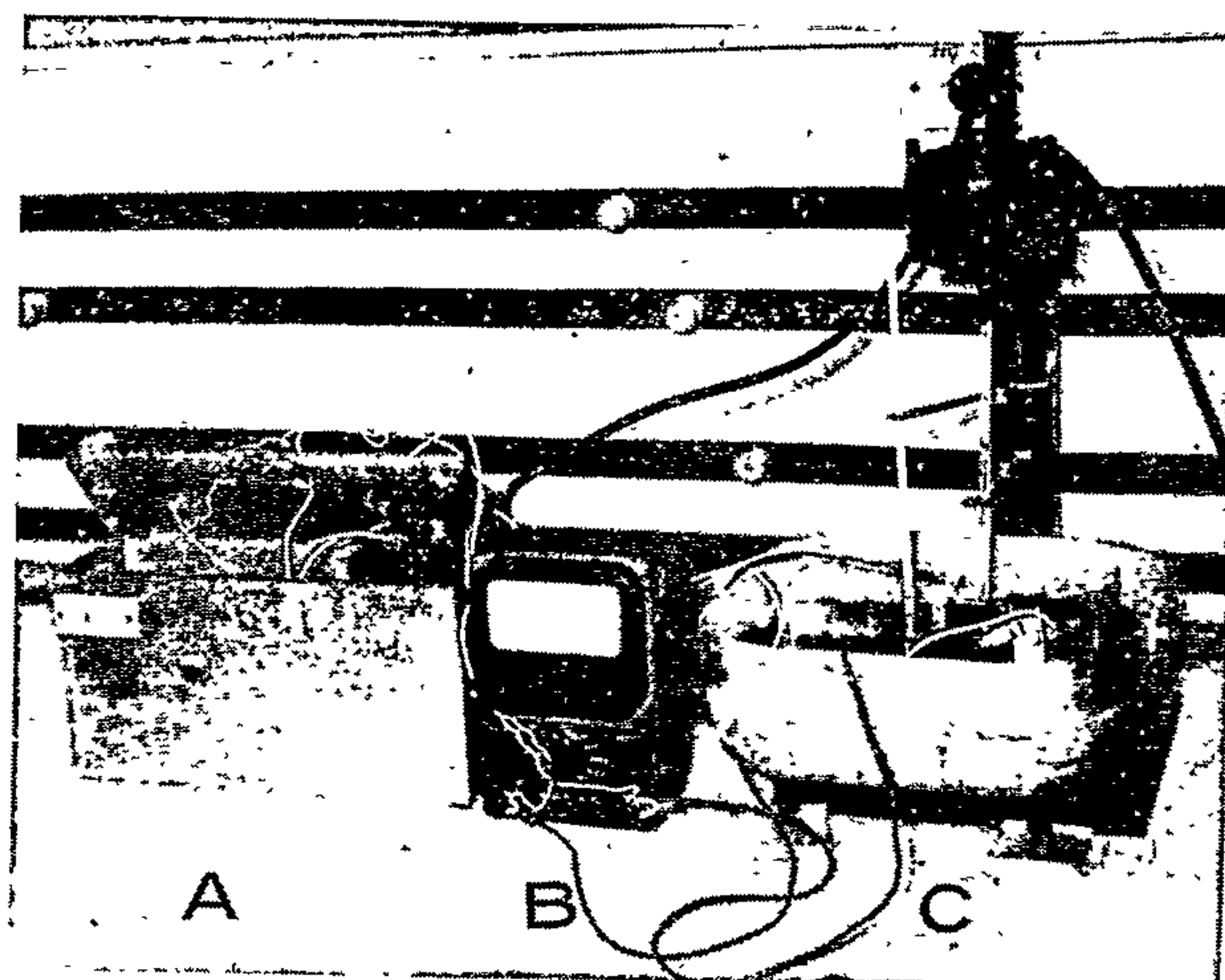


Fig. 5.—The equipment used for forming the gold matrix: (A) a unit containing a 2 V. storage battery and rheostat; (B) a multimeter; (C) the electroplating cell fitted with a stirrer immersed in a water bath.

PREPARATION OF THE SOLUTION

The solution was prepared as follows: 18.3 Gm. of potassium cyanide was dissolved in 500 ml. of distilled water at 60° C.; 14.1 Gm. of potassium gold cyanide was then dissolved in this, and 14.1 Gm. of potassium carbonate was added. Then, 11.4 Gm. of boric acid was dissolved in 150 ml. of distilled water at 60° C. and added to the solution. Distilled water was added to make a final volume of 1 L. The solution was allowed to stand overnight, then filtered prior to use. The entire operation was carried out under a hood because of the cyanide solutions.

OPERATING CONDITIONS AND EXPERIMENTAL RESULTS

The average current density employed was 3.2 amp. per square decimeter (30 amp. per square foot), and the temperature was maintained at 65° C. The voltage

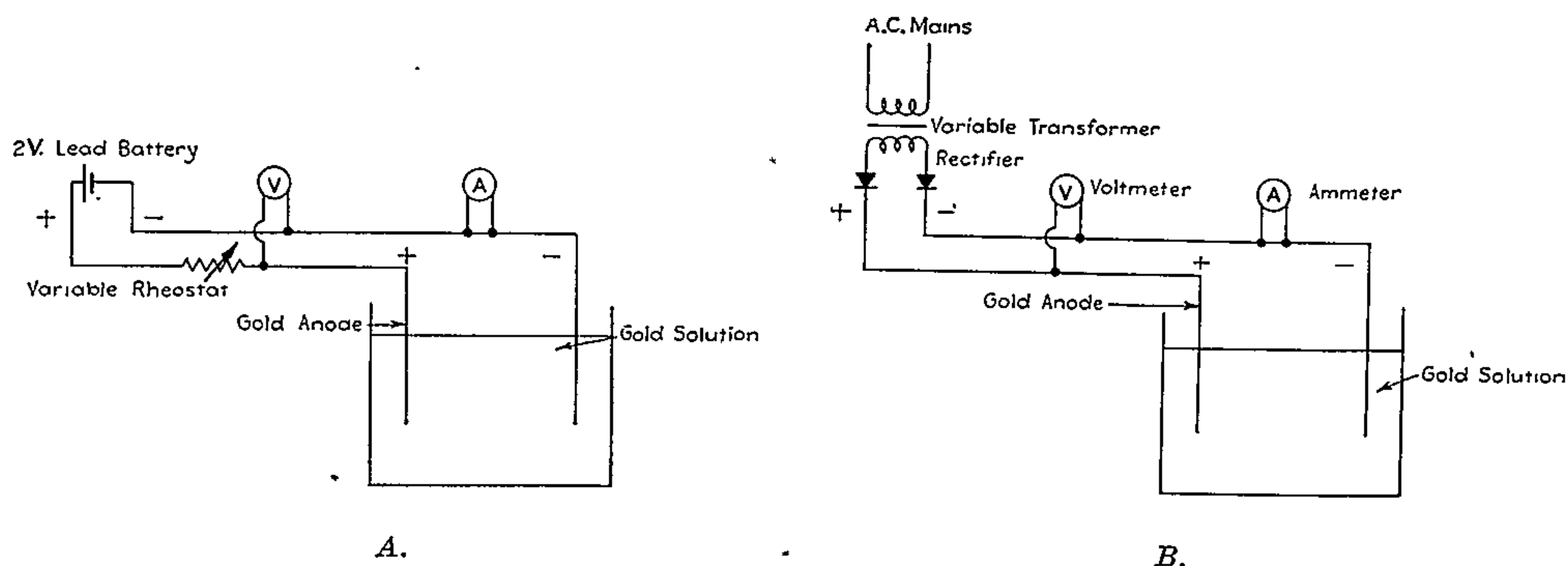


Fig. 6.—Diagrams of the electrical circuits. A, Circuit for forming the gold matrices. B, Circuit for experimental variations.

drop across the plating bath was 0.6 to 1 V. while the distance between the electrodes was 2 inches. The weight of the gold deposited per matrix was approximately 1 Gm., the average thickness of the deposit 0.005 to 0.006 inch. The throwing power of the solution was not measured, but the distribution of the metal was satisfactory (Fig. 7). When matrices were being produced, a current of 50 to 100 ma. was used for 1 to 2 hours, depending on the surface area and the shape of the metalized portion of the die. The deposited gold had a microhardness value of 64 (D.P.N. 25 Gm. load), while the value for annealed gold sheet was found to be 31 (Figs. 8 and 9). The electroformed gold presented no difficulties during subsequent manipulation and burnishing.

A series of experiments was conducted to determine the optimal conditions of operation. To facilitate the removal of the deposit, gold was plated on stainless steel cathodes having an area of 0.25 square inch. Temperature and current density were varied, and cathode efficiencies were determined. The hardness and microstructure of the deposits were examined (Fig. 10).

Smooth deposits of 0.004 inch thickness having a fine grain structure were obtained with a current density of 3.2 amp. per square decimeter in 1 hour at a temperature of 65° C. Current densities in excess of this led to the production of burned, powdery deposits (Fig. 11), while lower current densities produced deposits having a coarser grain structure. Interruption of the current supply caused the deposit to be laminated (Fig. 12). Smooth, fine grain deposits were produced at lower temperatures provided lower current densities were used, but this reduced the rate of deposition.

TABLE I. ELECTROPLATING SOLUTION

SOLUTION	GM.
Potassium gold cyanide	14.1
Potassium cyanide	18.3
Potassium carbonate	14.1
Boric acid	11.4
Distilled water	Quantity to make 1 L.

Fig. 7.—A cross section through a matrix. ($\times 10$; reduced 5/9.)



Fig. 8.—A section through a rolled gold anode. ($\times 150$; reduced $\frac{1}{3}$.) (D.P.N. 87.)



Fig. 9.—A section through an annealed rolled gold anode. ($\times 150$; reduced $\frac{1}{3}$.) (D.P.N. 31.)

At a temperature of 65° C., cathode efficiencies of 90 to 95 per cent were obtained. Anode efficiencies were above 100 per cent when the anode current density was below 0.86 amp. per square decimeter. When the anode current density exceeded this value, the anode efficiency was as low as 10 per cent although polarization films were not visible on the anode surface.²⁰ In order to prevent a reduction in the gold concentration of the solution which would occur from these conditions, the anode area was maintained at four times that of the cathode area so that the anode current density was below 0.86 amp. per square decimeter.

DISCUSSION

The pH of the solution during these experiments was constant at a value of 10.2 (electrometric), and the free cyanide concentration, which was determined at regular intervals, was maintained at 10 Gm. per liter by periodic additions of potassium cyanide. Distilled water was used to maintain a constant solution level. During one series of experiments, 14.2 Gm. of gold was deposited from 1 L. of solution and 16.5 Gm. of gold was dissolved from the anode. Thus, not allowing for dragout, the gold concentration of the solution had increased, and the deposit produced was still satisfactory. The use of a larger volume of solution would ensure a more constant concentration of metal, but this would be uneconomical. In the solution described, gold anodes could be replaced by insoluble anodes in the production of two or three matrices in every fifteen.

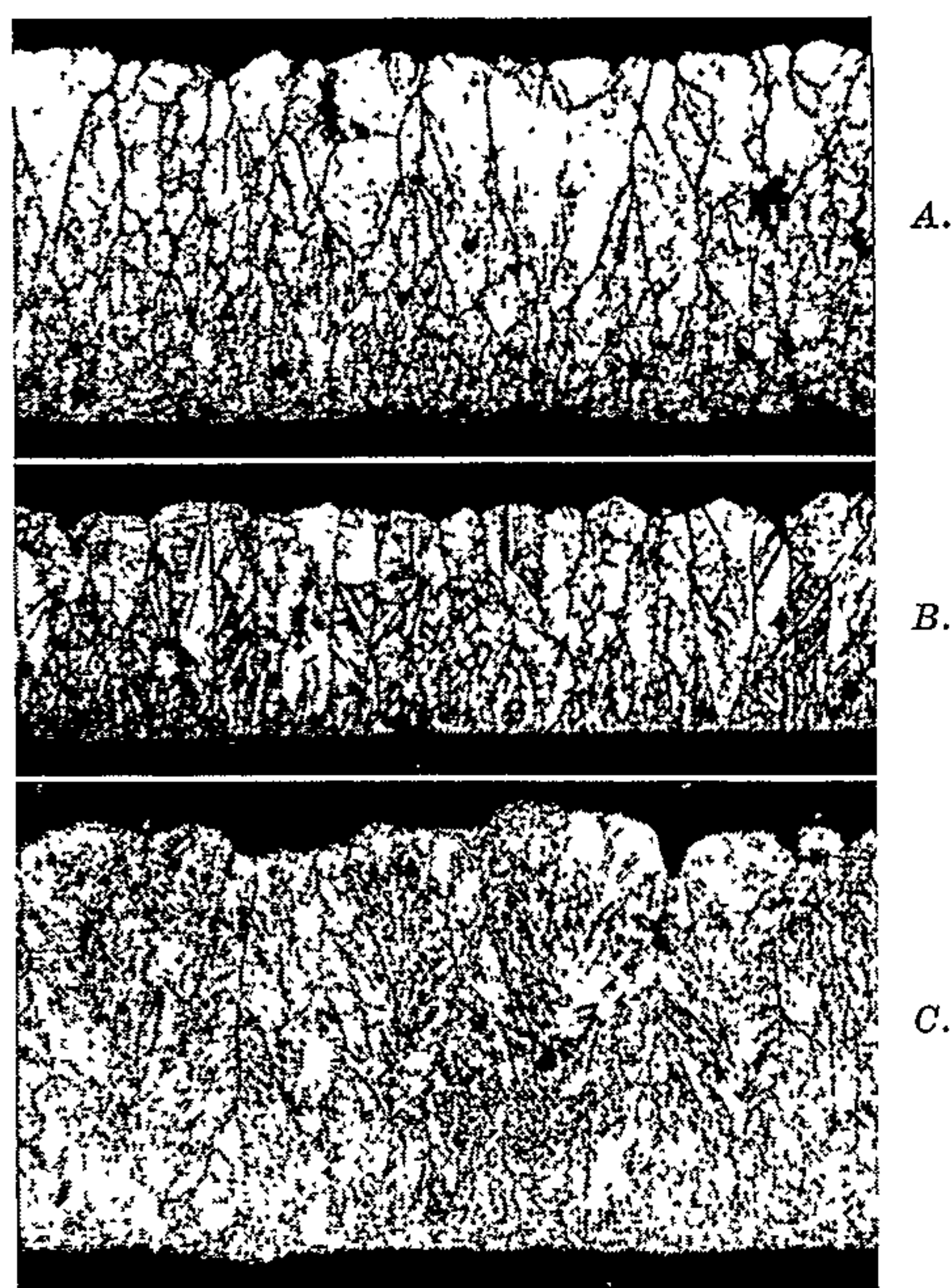


Fig. 10.—Sections through deposited gold showing the effect of current density. *A*, Current density 0.86 amp. per square decimeter (8 amp. per square foot); temperature 65° C.; mean D.P.N. 53. *B*, Current density 1.5 amp. per square decimeter (14.4 amp. per square foot); temperature 65° C.; mean D.P.N. 64. *C*, Current density 3.2 amp. per square decimeter (30 amp. per square foot); temperature 65° C.; mean D.P.N. 64. ($\times 500$; reduced $\frac{1}{3}$.)

Fig. 11

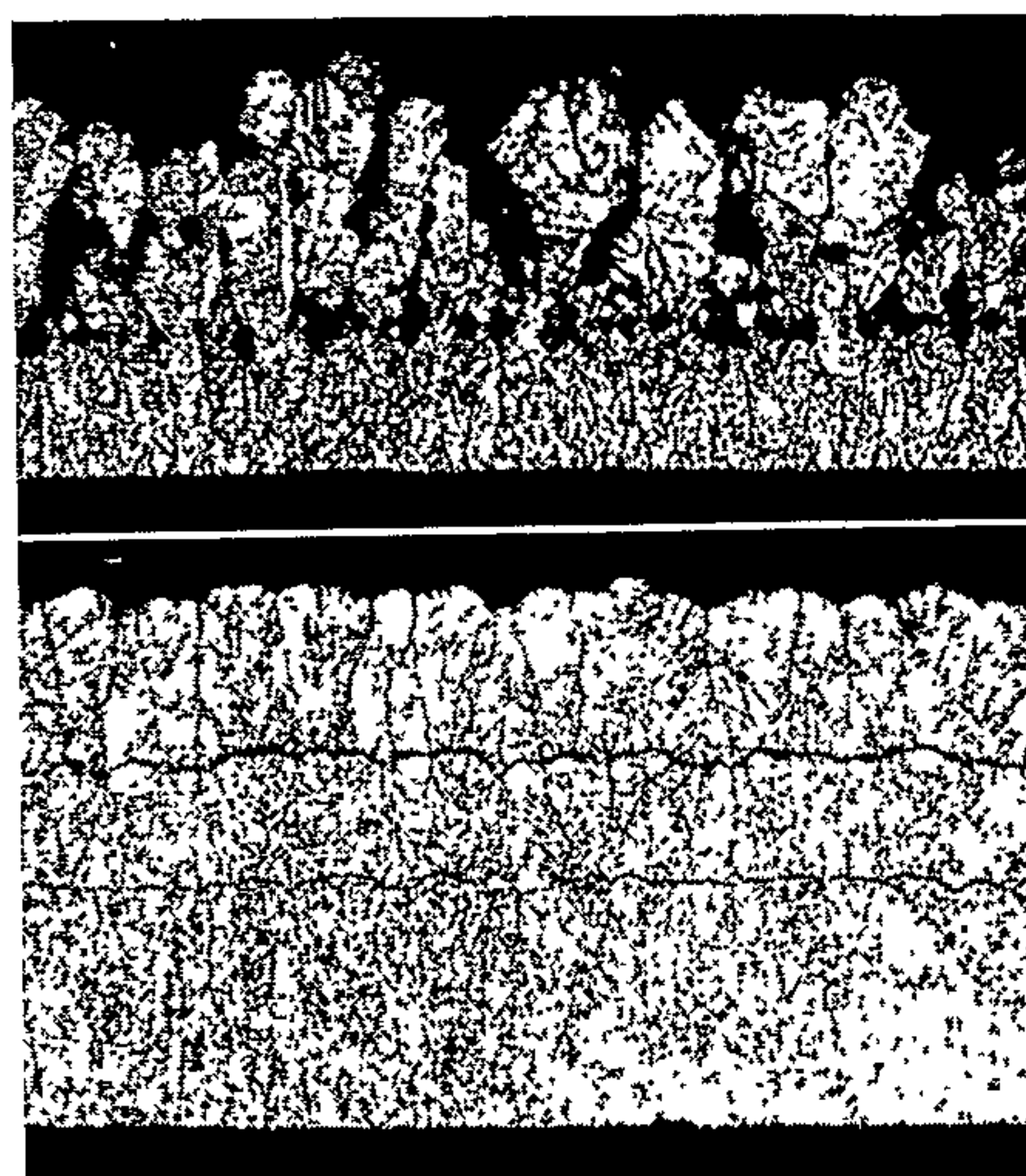


Fig. 12

Fig. 11.—A section showing the effect of excessive current density, 5.3 amp. per square decimeter (50 amp. per square foot); temperature 65° C. ($\times 500$; reduced $\frac{1}{3}$.)

Fig. 12.—A section showing the effect of current interruption. ($\times 500$; reduced $\frac{1}{3}$.)

For the successful operation of this solution, it is necessary to control the metal and the free cyanide concentrations. However, the life of the solution depends upon the accumulation of carbonates. When present in excess, carbonates which are formed by the oxidation of cyanide cause a loss of cathode efficiency, and the deposits obtained are unsatisfactory.

SUMMARY

A thick gold matrix has been produced by electroforming. Using the gold cyanide solution described, the matrix can be produced in 1 to 2 hours, the rate of deposition being 0.004 to 0.005 inch per hour at a temperature of 65° C. and a current density of 3.2 amp. per square decimeter.² This matrix can be adjusted in detail to the prepared cavity and the inlay completed by casting a suitable gold alloy into the matrix. The cast gold apparently forms an intermediate alloy with the pure gold matrix. The behavior of the electroplating bath and of the electrodes and the use of additive agents are being further investigated. Clinical applications are being investigated, and studies are in progress to produce an electroformed matrix that has a high melting point for use in ceramic restorations.

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ELECTRODEPOSITED COPPER INDIVIDUAL IMPRESSION TRAYS

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ALL IMPRESSION materials exhibit some dimensional change on setting.¹ The greater the amount of impression material in a given region, the greater this change, and the greater the distortion.^{2,3*} To minimize this factor, an equal distribution of the impression material is essential between the tray and the oral structures being recorded. For example, if an improperly fitting tray permits an excessive thickness of impression material on one side of the dental arch, then a greater change in dimension and distortion will occur in this region.

Most impression materials shrink on setting. Because the material is locked firmly to the surface of the tray and artificial stones have a slight setting expansion, an oversized cast results.^{2,4*} If the shrinkage of the impression material is uniform, it can be calculated and compensation can be made for it, usually during the casting procedure. However, if the cast is also distorted, then the principle of compensation is defeated. In order to control the amount and distribution of the impression material, it is essential to use an impression tray made to the requirements of the individual patient.

Metal trays are generally used for preliminary impression procedures while acrylic resin trays are recommended for individual or final impression procedures. The basic claim to popularity of the resin tray is that it is easy to make.⁵⁻⁸

With this in mind, a simplified technique to produce an individual metal tray, nonperforated or perforated as required, was investigated. The procedure involved the preforming of a tray in annealed soft brass mesh, and strengthening the mesh by the electrodeposition of copper.

FORMATION OF THE MESH

The preliminary cast was covered with a uniform thickness of baseplate wax to provide space in the tray for the final impression material (Fig. 1). A 3 mm. thickness of wax is recommended.^{9,10}

The size of the perforations in the tray is an important factor controlling the accuracy of elastic impression materials. When the perforations are too large or too numerous, the light pressure needed to bring the impression material into intimate contact with the tissues will cause an excess of the material to extrude through the perforations.³ The size of the perforations in an electroplated tray is de-

*Hollenback, G. M.: Personal communication.



Fig. 1.—A uniform thickness of baseplate wax is added to the diagnostic cast to form a spacer for the impression tray.

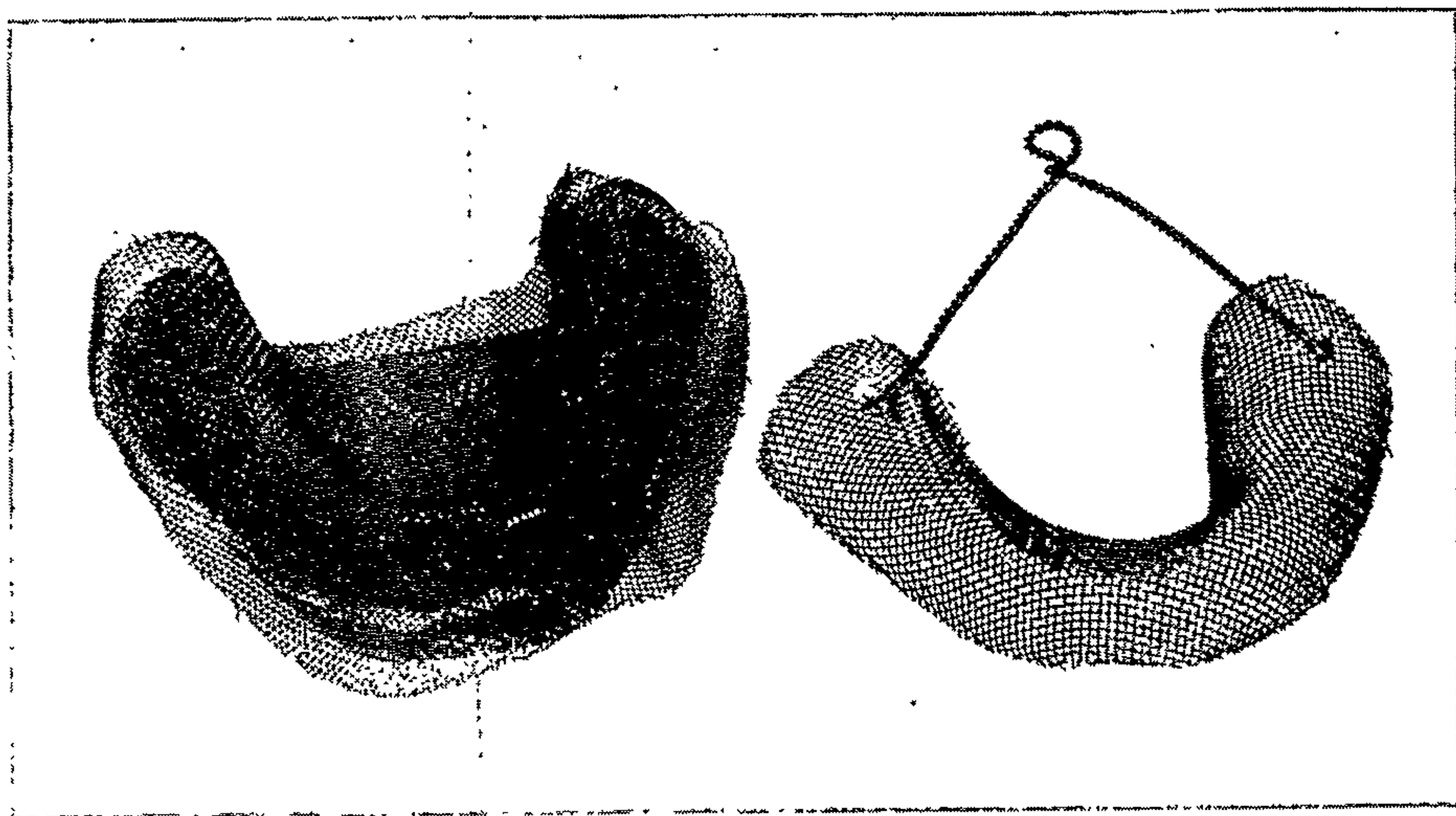


Fig. 2.—A, The annealed brass mesh is contoured to the waxed cast. The border of the tray is outlined in pencil. B, The mesh is cut to the desired shape, and the wire is soldered preparatory to electroplating.

pendent upon the size of the brass mesh and the amount of copper deposited on the mesh.

Perforations that are similar in size to those in a stock hydrocolloid tray were produced by 16 mesh 28 gauge brass while 30 mesh 33 gauge brass produced a non-perforated tray after the interstices were completely filled by electrodeposition of copper. A tray constructed with 24 mesh 30 gauge brass provided perforations of intermediate size (See Figs. 6 to 8).

The selected mesh was annealed by heating it in a Bunsen flame, and allowing it to cool. This treatment also burned off the oil coating that had been applied to the brass mesh during its manufacture. Oil is an electrical nonconductor and its removal is essential for the successful electrodeposition of copper. The annealed mesh

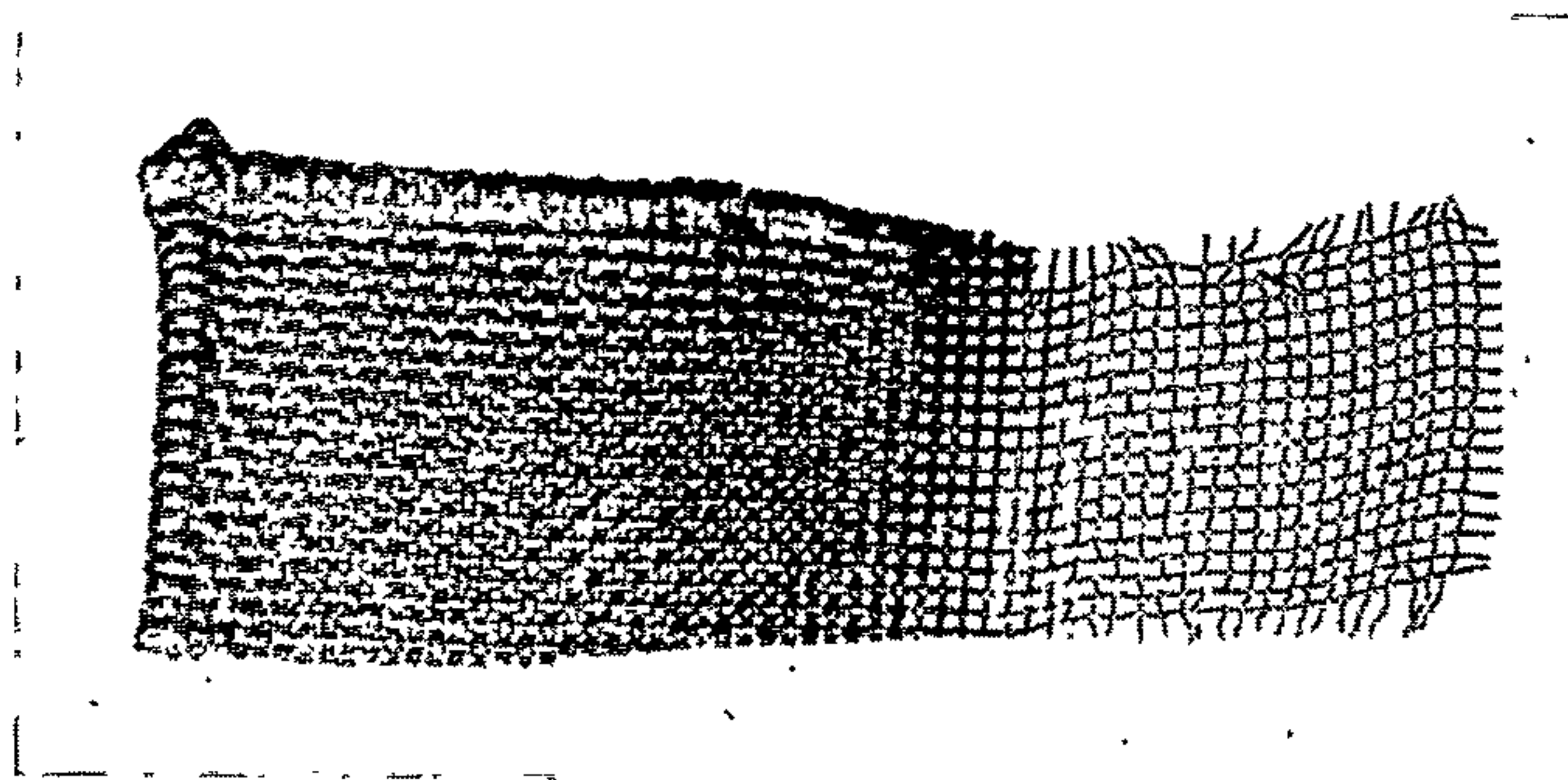


Fig. 3.—A section of mesh after deposition of copper shows the copper built up at the edges. The lower border has been cut away for thickness comparison tests.

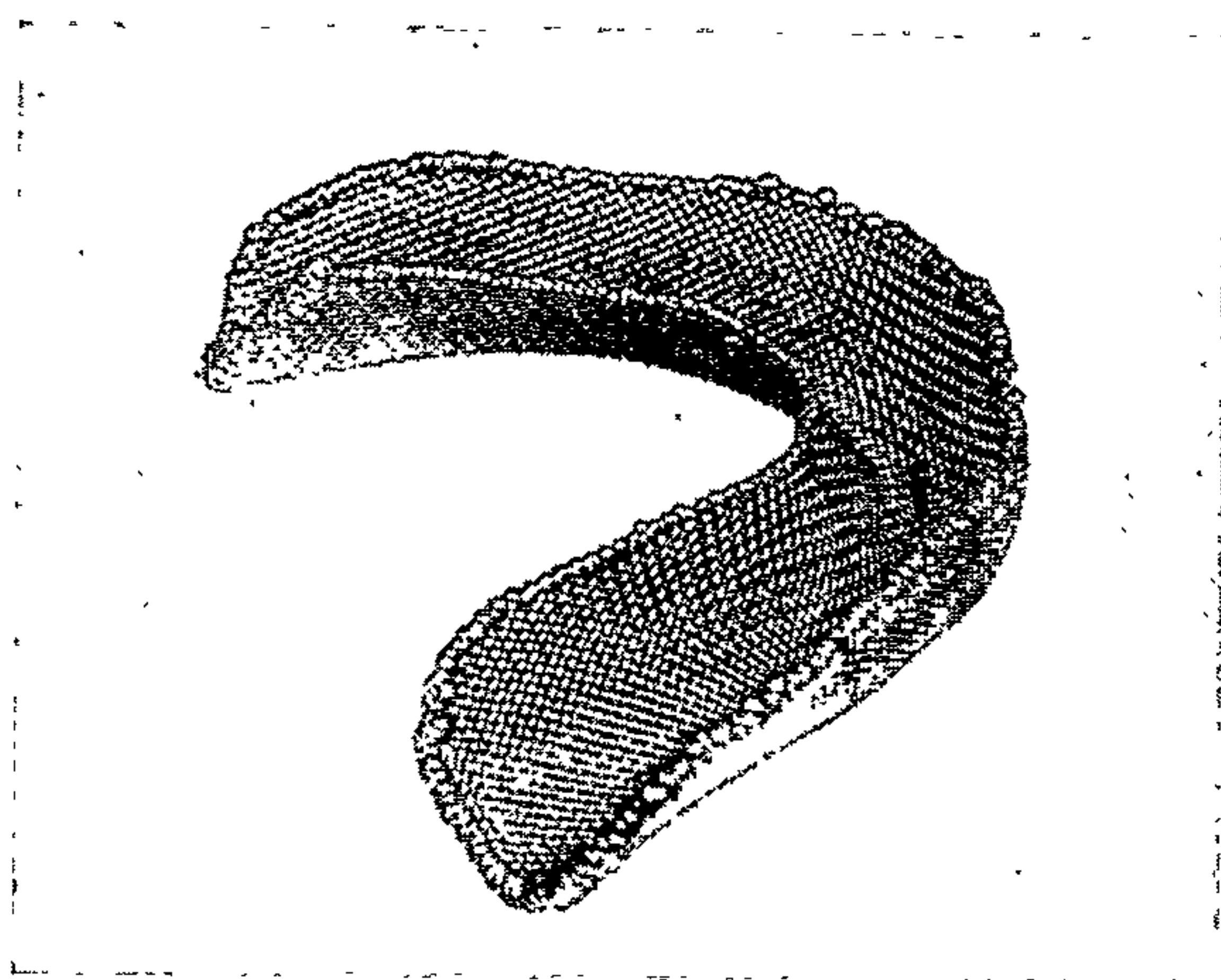


Fig. 4.—The mesh as shown in Fig. 2,B, after deposition of copper. The conductive wire has been removed, and the tray is ready for adjustment of the border.

was adapted to the wax on the cast, and the desired form of the tray was marked in pencil on the mesh (Fig. 2,A). The mesh was removed, trimmed, and readapted to the cast. Then, a rigid copper wire was soldered to the posterior borders to reinforce the annealed mesh and to prevent accidental damage or distortion to the tray during subsequent processing (Fig. 2,B).

The mesh was again heated in a flame to burn off residual wax and then strengthened by applying a copper deposit of 0.01 to 0.02 inch in thickness.

DEPOSITION OF COPPER

An electroplating solution made up of 200 Gm. of copper sulphate, 50 Gm. of sulphuric acid, and 1 L. of water gave satisfactory results. The cathode current density was set at 15 to 20 amp. per square foot with an operating temperature of 15 to 24° C. The distance between electrodes was 7 to 9 inches and the voltage across the tank was maintained at 2 to 4 v.

The solution was placed in a polyethylene tank with a capacity of 9 L. The

anode area was three times that of the cathode. Current was supplied by a single phase 6 v. selenium rectifier with an output of 25 amp. Increased operating temperatures, the use of agitation, and addition of various agents increase the rate of deposition of copper. However, increased control of the solution is required when increased deposition is desired.

The electroplating solution requires little maintenance during operation other than occasional additions of water to compensate for evaporation. Copper anodes were placed on either side of the brass mesh cathode to provide a uniform distribution of copper over the surface. However, the concentration of current was greater on the edges than in the center of the cathode and so the deposit of metal was greatest on the edges of the copper mesh (Figs. 3 and 4). The rough edge was completely covered over with a deposit of copper and this was ground away to form a smooth border.

The current density was varied according to the area of the mesh. The time required for deposition of the metal varied according to the amount of copper required between the interstices of the brass mesh. The trays were produced at a current density of 15 to 20 amp. per square foot for a period of eight hours.

The cathode was assumed to be a flat object and both sides were included when determining current density. For example, if the area of the mesh was 6 square inches, the total surface area was considered to be 12 square inches, and if a current density of 18 amp. per square foot was required, the amperage necessary was $18 \times \frac{12}{144}$ or 1.5 amperes. The electrical circuit diagram is seen in Fig. 5.

FINISHING THE TRAY

The electroplated tray was thoroughly washed to remove any traces of electrolyte, and the wire used for electrical contact was removed. The rough edge of the mesh which was built up by electrodeposition of copper was ground smooth and polished (Fig. 4). The tray was then electroplated in a bright nickel bath for 5 to 10 minutes to prevent tarnishing (Figs. 6 to 8). The inner surface of the border can be left over contoured if a rim-lock type of tray is preferred.

When handles are desired, they should be made from copper, brass, or bronze

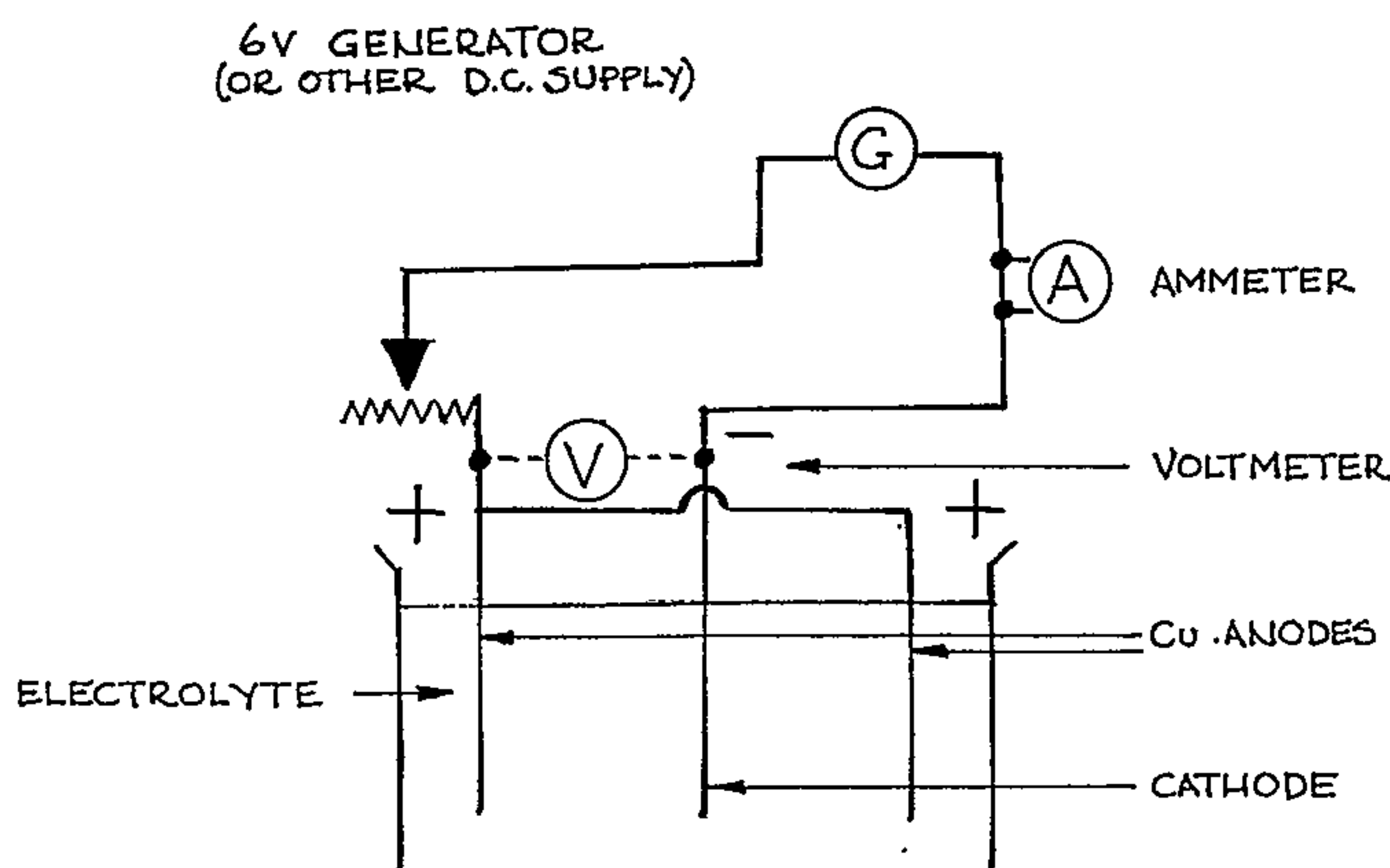


Fig. 5.—The circuit diagram for the electroplating instrumentation.

and attached prior to the nickel-plating procedure. After removing oxides and grease, the tray and handle should be scratch brushed, particularly where soft solder has been used.

DISCUSSION

The dimensional stability of an impression cannot be greater than the tray in which it is made. Metal trays have greater rigidity and stability than resin trays. According to Skinner and Phillips,³ 3 to 5 per cent of self-cured resin is free monomer which may cause subsequent distortion. Studies with plastic bands and trays have shown that due to water absorption the possibility of distortion is increased.²

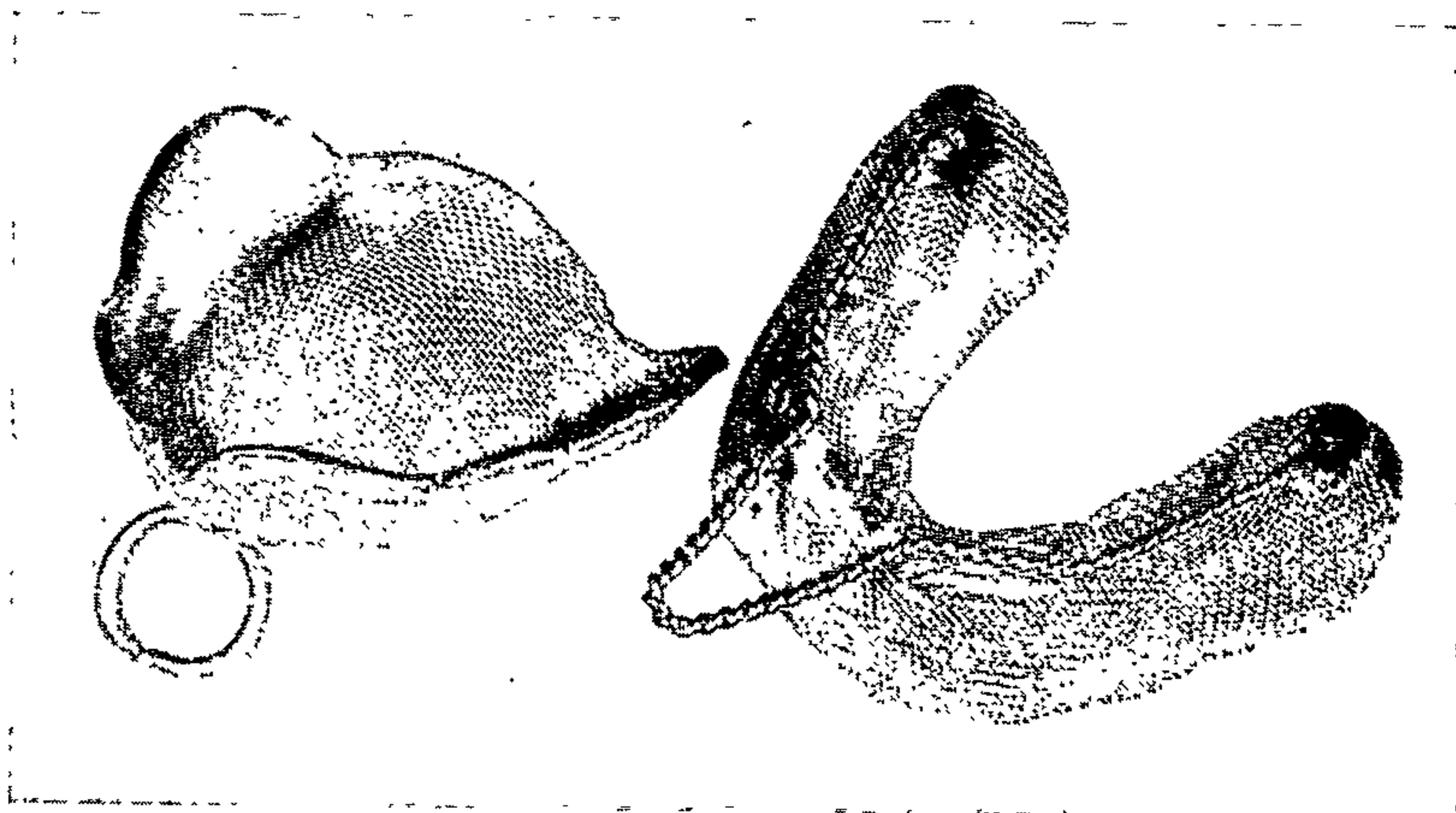


Fig. 6.—Handles have been added to the completed trays and a final coat of nickel has been applied. The interstices of the brass mesh (30 mesh 33 gauge) are completely filled so as to produce a nonperforated tray.

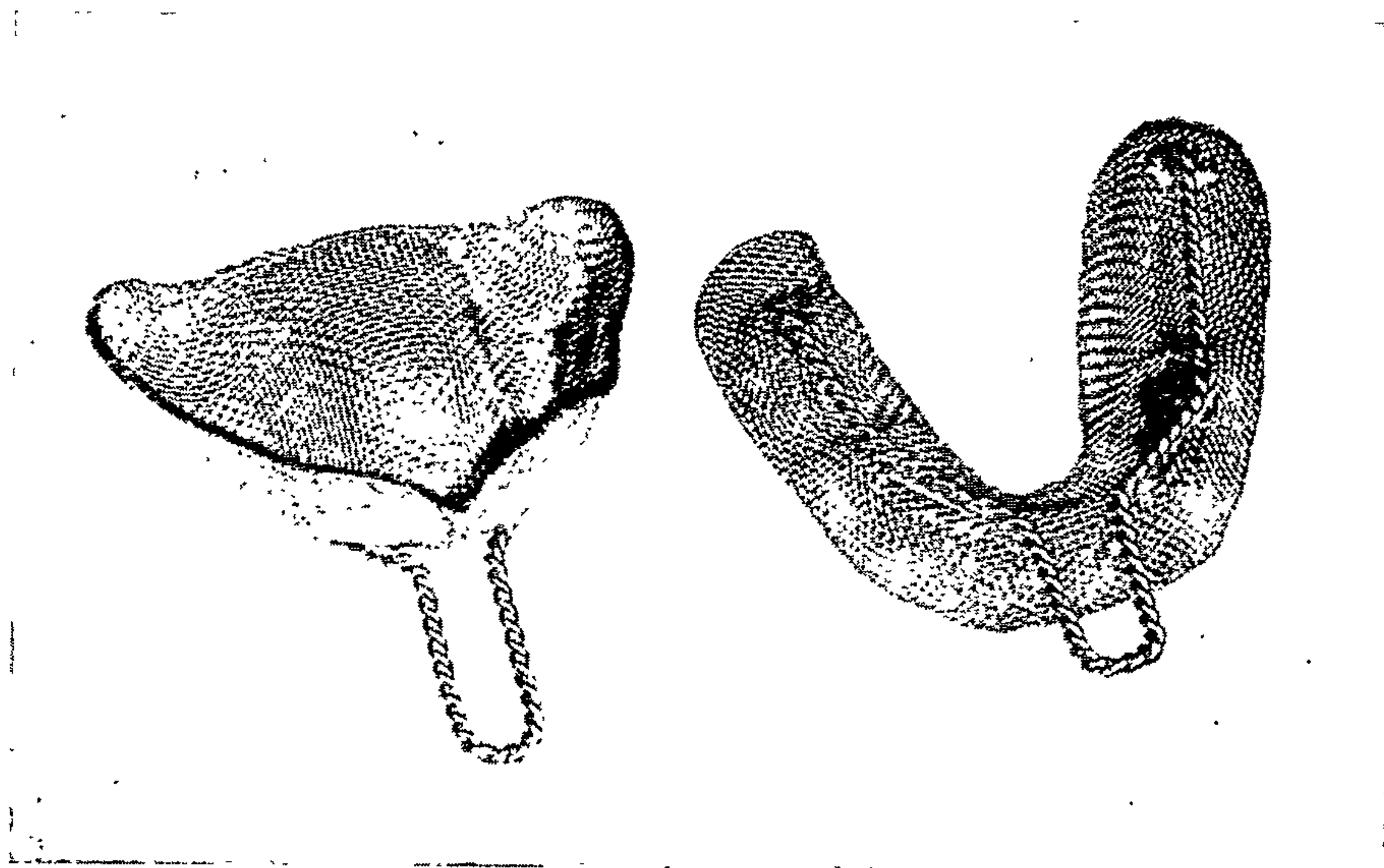


Fig. 7.—The brass mesh (24 mesh 30 gauge) has been partially filled to give small-sized perforations in the completed trays.

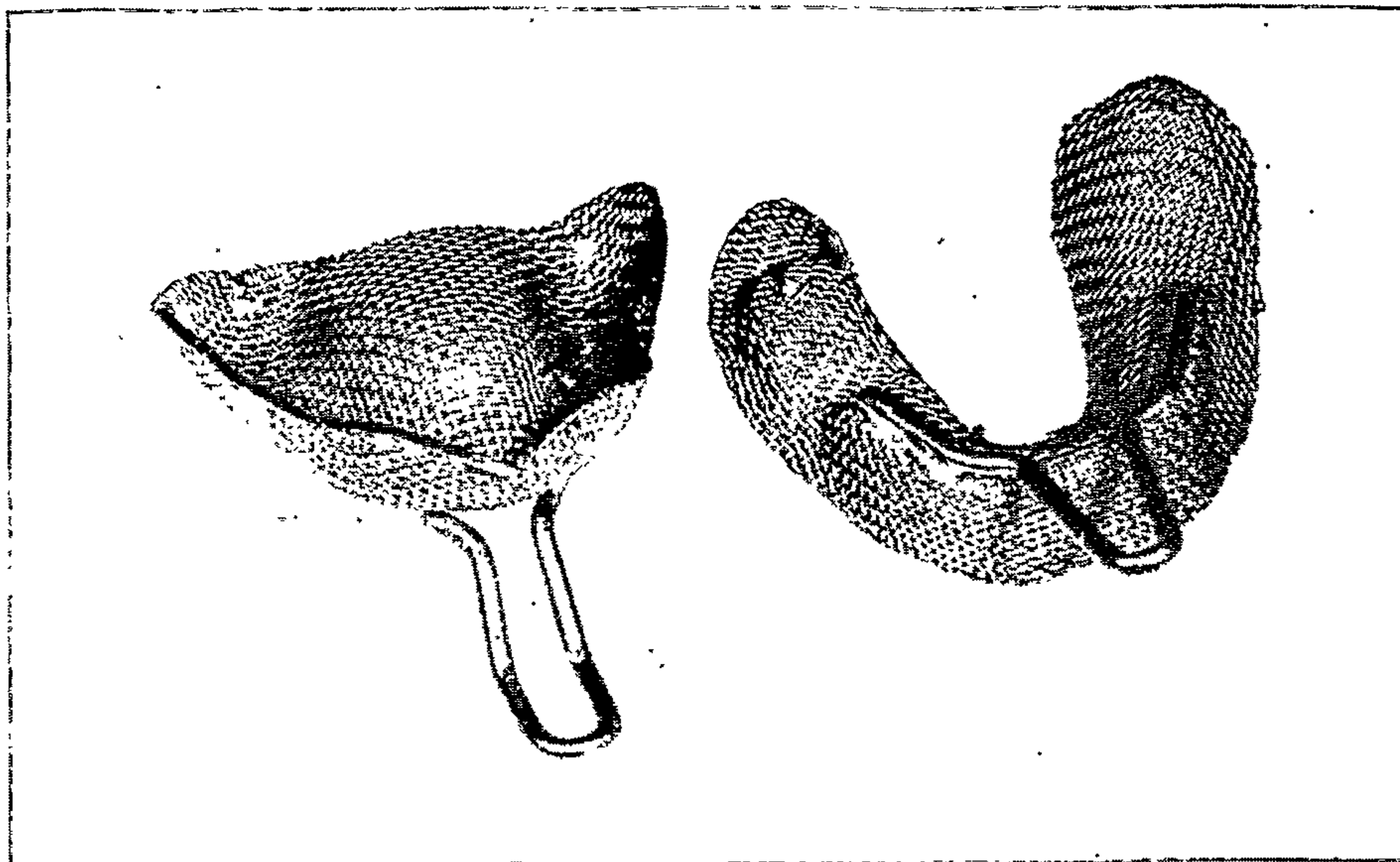


Fig. 8.—The 16 mesh 28 gauge brass mesh provides larger perforations than seen in Fig. 7. By selection of the mesh size and the amount of copper deposited, the diameter of the perforations can be controlled.

The coefficient of thermal expansion for acrylic resin is approximately 4 to 5 times that of metals used for trays and might provide an additional factor to control in extreme circumstances. Acrylic resin trays are difficult to sterilize. However, metal trays can be sterilized by autoclaving or boiling.

The electroformed mesh technique was developed to simplify the construction of metal trays. The size of the tank and the current supply can be modified to permit concurrent production of a large number of trays. For example, the power output of the equipment used for these experiments (25 amperes) could produce about 16 trays in an eight hour period. Since little supervision is necessary, the unit could be used on a 24 hour basis.

Although the construction of individual trays for impressions for operative dentistry or fixed and removable partial dentures has been stressed, trays for final impressions for complete dentures, or stock trays can be produced. A protective coating of bright nickel is recommended, if the tray is to be used on different occasions.

SUMMARY

Individual metal impression trays have been produced by strengthening a pre-formed brass mesh by electrodeposition of copper. The size of the perforations in the tray are variable and are determined by the mesh selected and the thickness of copper deposited. A fine mesh is used for making a nonperforated tray, so that the interstices are completely filled by electrodeposition of copper.

I would like to thank B. W. Armstrong, A.S.T.C. (Chem. Eng.), A.R.A.C.I., Senior lecturer, School of Metallurgy, University of New South Wales, for his assistance in the preparation of this article.

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Gold-plating of cobalt-chromium*

O. W. Rogers

Introduction

One of the most significant factors influencing removable partial denture construction has been the advent of cobalt-chromium alloys. Because of their high strength, hardness, lightness in weight and resistance to tarnish and corrosion, they have generally supplanted the use of gold alloys, but this has chiefly been due to an economic factor rather than advantages in physical properties. The bright silver colour of cobalt-chromium is often unacceptable to the patient and, to improve the aesthetics, visible clasps are often gilded by flashing with gold solder.

The development of electrodeposited hard bright golds offers an alternative method of gilding which can be applied to individual clasps, or to the entire casting.

The use of cobalt-chromium, as with other chromium containing alloys such as stainless steel and nichrome, is based on its high

resistance to corrosion, particularly in the oral environment. Chromium has a passivating effect due to a thin oxide layer which forms on the surface of the metal. This oxide layer is continuous, non-porous and self-healing. However, passivation is not a constant state—it exists only in certain environments and under certain conditions. For example, these conditions are changed in soldering when a flux is used that dissolves the surface oxides.⁽¹⁾

Surface activation

To ensure intimate contact between chromium containing alloys and the metal being deposited the surface film must be removed and prevented from reforming.

Various methods are available for activating stainless steel to remove the passive surface and permit intimate metal to metal contact during electrodeposition.⁽²⁾ Because of the

* The author is indebted to Mr. B. W. Armstrong, Senior Lecturer at the School of Metallurgy, University of New South Wales, and the staff of the Commonwealth Bureau of Dental Standards, for their help in the preparation of this paper.

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⁽¹⁾ Bien, S. M., and Ayers, H. D.—Autoradiographs of solder joints with fluorine. *J. D. Res.*, 38: 3, 428-435 (May-June) 1959.

⁽²⁾ Bien, S. M., and Ayers, H. D.—Soldered joints on plated rustless alloys. *J. D. Res.*, 37: 2, 368-372 (Apr.) 1958.

similar nature of the passive surface on stainless steel and the cobalt-chromium alloys, experiments were conducted to determine if the cobalt-chromium alloys would respond to similar activating processes⁽³⁾ prior to gold-plating.

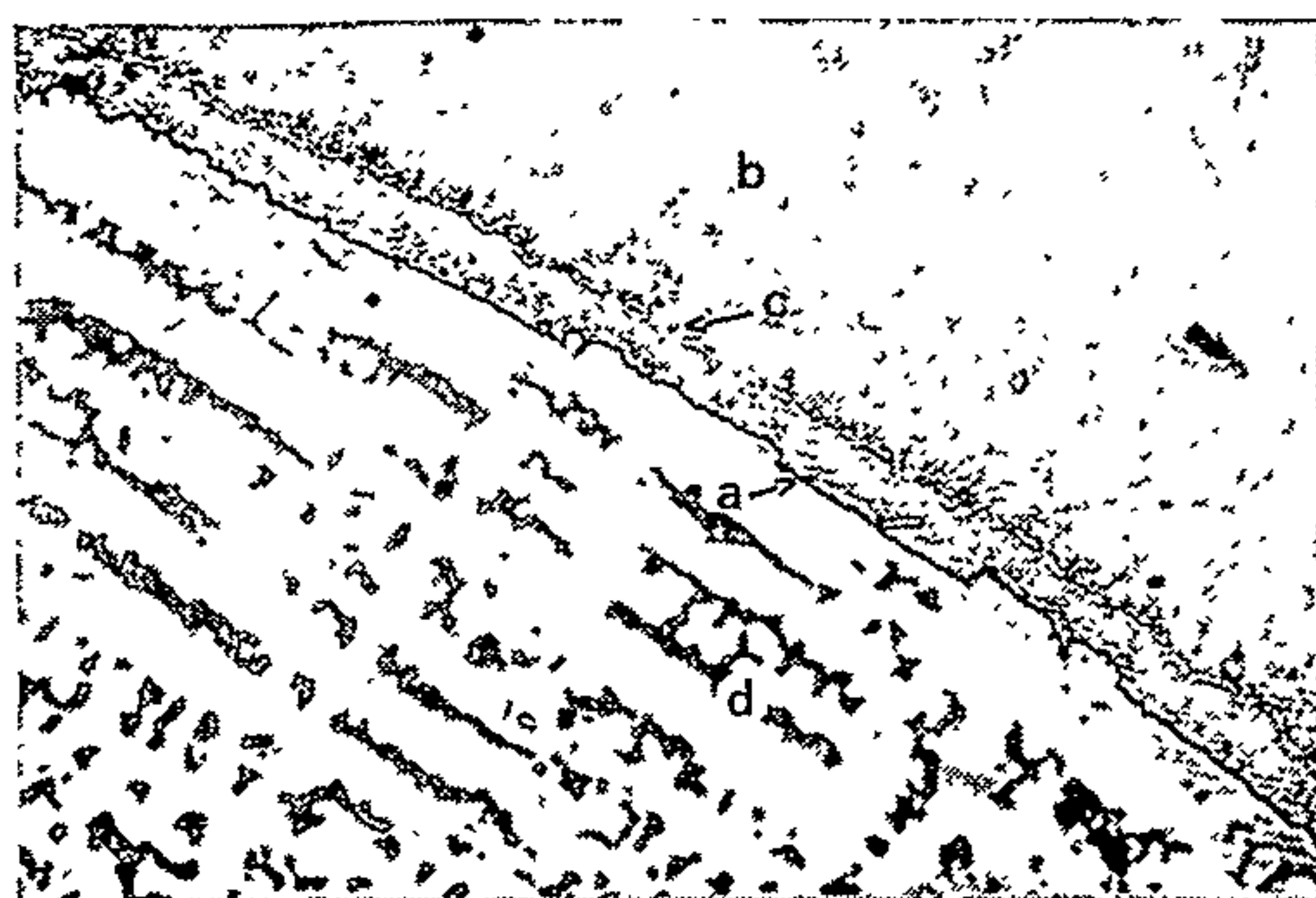


Fig. 1.—The interface between electroplated gold and cobalt-chromium. (a) Interface showing the keying effect of anodic etching on cobalt-chromium; (b) electrodeposited gold; (c) line of current interruption in electroplated gold; (d) cobalt-chromium alloy. ($\times 240$.)

Activation treatment in sulphuric acid at concentrations between 5 per cent and 50 per cent by volume, and at current densities between 0.1 and 2.1 amp./dm.² did not produce consistent results. Immersion in Wood's nickel electrolyte for 15 minutes followed by the application of a current of 3.2 amp./dm.² did not produce a good coherent bond.

The cobalt-chromium alloys were successfully treated in Wood's nickel electrolyte when an anodic current density of 3.2 amp./dm.² was used for two minutes followed by cathodic treatment at 3.2 amp./dm.² for six minutes. This electrolyte is a low pH solution with a low metal iron concentration. As the metal cathode efficiency is very low, copious evolution of hydrogen occurs simultaneously with the deposition of nickel. The anodic treatment removes the oxide film and activates the surface of the cobalt-chromium alloy. The cathodic treatment has a reducing effect due to the hydrogen released. This prevents the reformation of the oxide film and creates a chemically clean surface for the deposition of nickel.

When the surface of the casting is covered with nickel the casting can be electroplated with gold.

⁽³⁾ Australian standard code of recommended practice for the preparation of metal surfaces for electroplating. A.S. CK13-1966.

Materials and method

The Wood's nickel electrolyte⁽⁴⁾ used in the activation process had the following formula:

Nickel chloride	240 g.
10 N hydrochloric acid	86 ml.
Distilled water	to 1 L.

Eight litres of the solution are placed in a PVC tank and operated at a temperature of 21-26° C. A nickel electrode is used. No agitation is necessary.

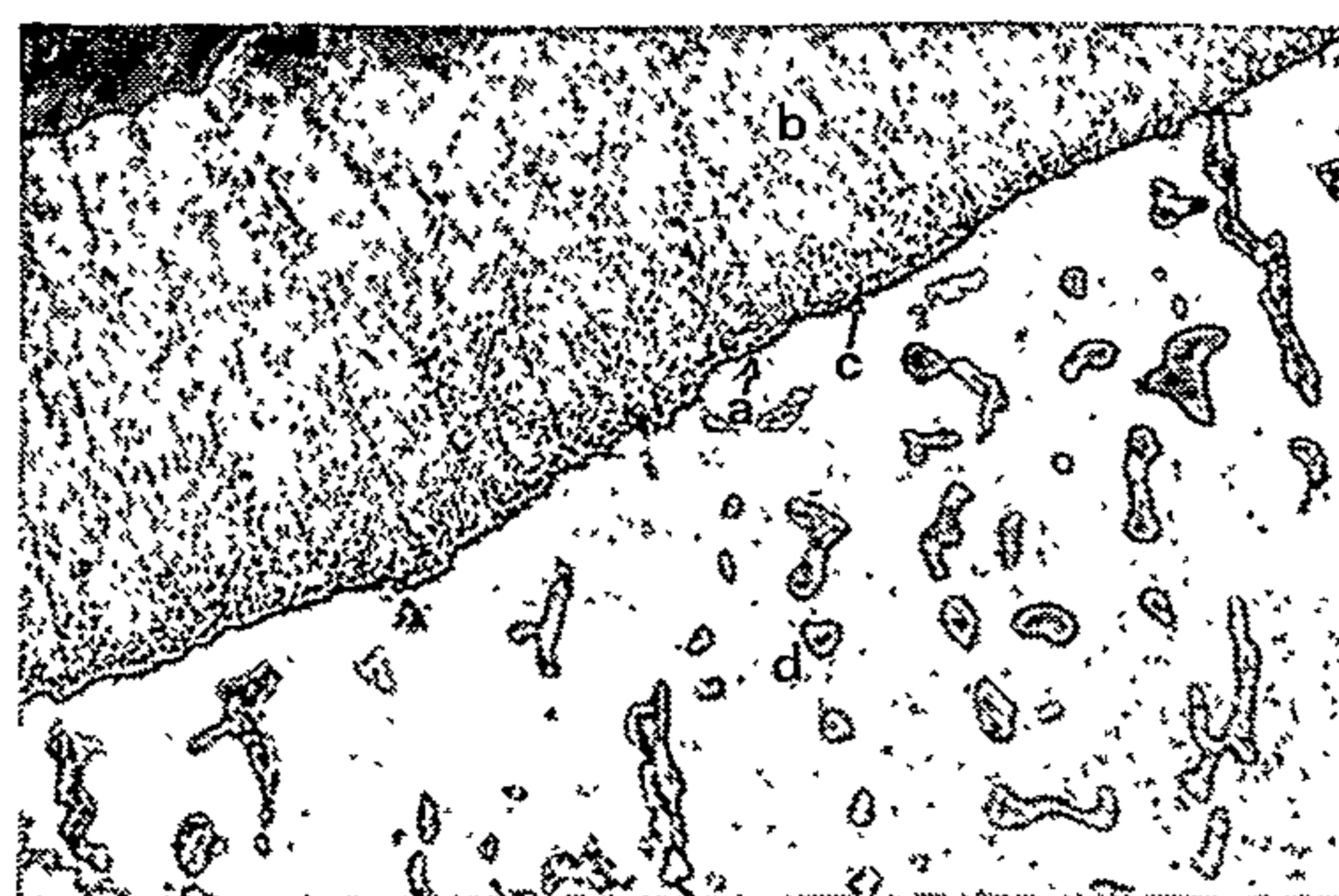


Fig. 2.—The interface between electroplated gold and cobalt-chromium. (a) Interface showing irregularity due to anodic etching but keying effect is less evident; (b) electrodeposited gold; (c) line of current interruption in electroplated gold; (d) cobalt-chromium alloy. ($\times 240$.)

A copper wire is attached to the saddle area of the casting to simplify handling and provide electrical contact during the activation and plating processes.

The casting is thoroughly cleaned in a proprietary alkaline metal cleaning solution and then washed in water.

After this treatment the casting is placed in the Wood's nickel electrolyte and given anodic treatment for two minutes at 3.2 amp./dm.². The current is then reversed—the casting now being the cathode—and the same current density (3.2 amp./dm.²) applied for six minutes. The casting is removed, rinsed and then immediately gold-plated by one of the conventional methods.

If only a part of the cobalt-chromium casting, such as the clasps, are to be plated the remainder of the casting is masked off after

⁽⁴⁾ Blum, W., and Hogaboom, G. B.—Principles of electroplating and electroforming. New York, McGraw-Hill, 3rd ed., 1949 (p. 129).

cleaning by using a non-conductive varnish. The saddle area is left unvarnished to maintain a suitable electrical contact for the copper wire.

Results

Photomicrographs, Fig. 1, 2, show electroplated gold deposits on cobalt-chromium. The interface (a) is irregular and notched by the electrolytic etching. This could have an additional keying effect upon the electro-deposited metals. The nickel deposit is not apparent in the photomicrographs as its estimated thickness is only 6×10^{-6} inch.

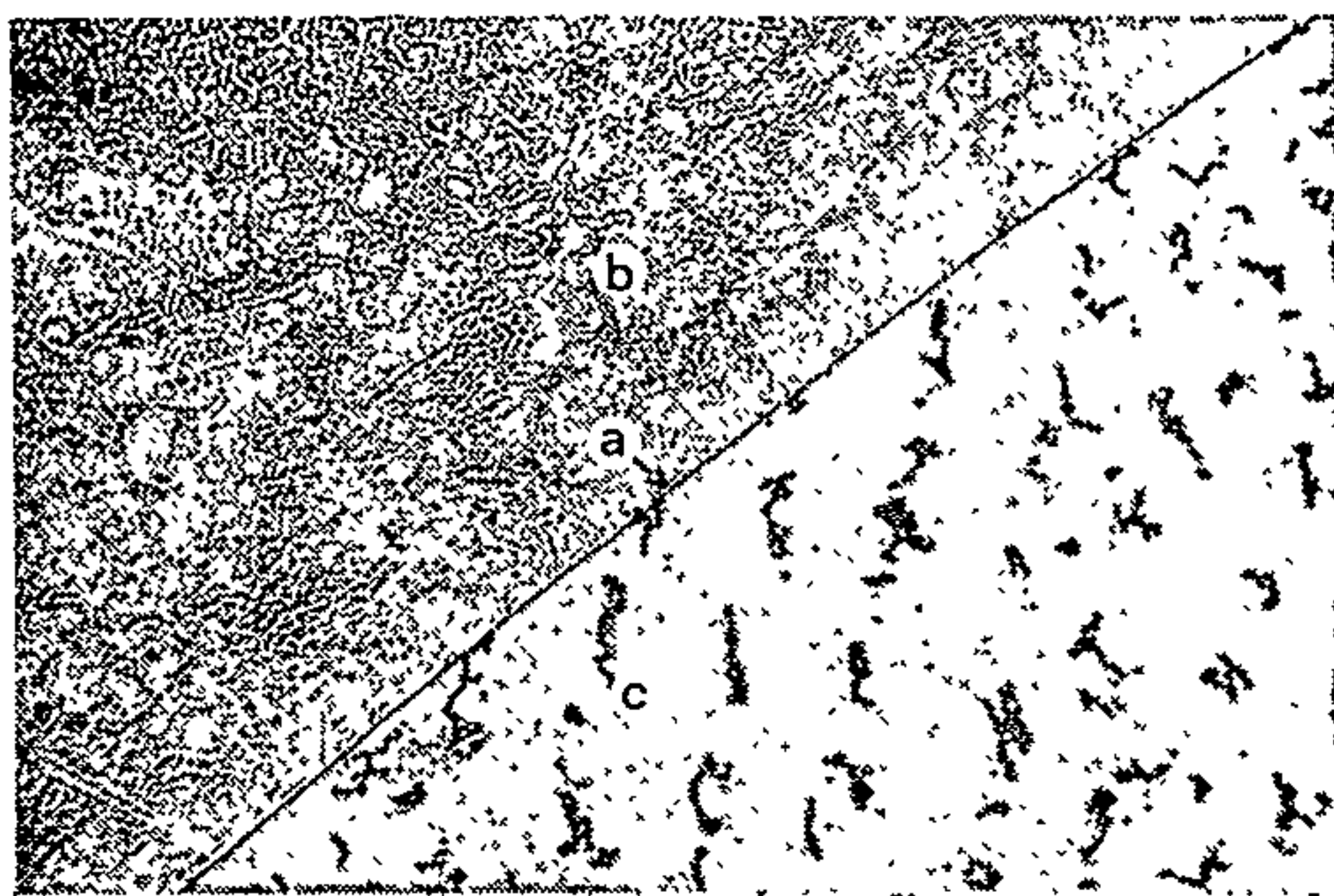


Fig. 3.—The interface between 18 ct. gold solder and cobalt-chromium. (a) Interface is straight and shows no irregularities; (b) 18 ct. gold solder; (c) cobalt-chromium alloy. ($\times 240$.)

The effect shown at line (c) is due to current interruption during electrodeposition of the gold. Passivation occurred when the casting was removed for inspection after gold-plating commenced to check the continuity of the gold deposit. This effect has been noted before⁽⁵⁾ and has no clinical significance.

Figure 3 is a photomicrograph of the interface between cobalt-chromium and 18 ct. gold solder. The interface (a) shows no irregularity or keying.

* Sel-Rex B.D.T. Gold Process.

- ⁽⁵⁾ Rogers, O. W., and Armstrong, B. W.—Electroforming a gold matrix for indirect inlays. *J. Pros. Den.*, 11:5, 959-966 (Sept.-Oct.) 1961.
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Discussion

The durability of the plating will depend upon the hardness of the gold deposited and its thickness. Gold electroplated from conventional cyanide electrolytes has a hardness up to 70 Knoop.⁽⁵⁾⁽⁶⁾⁽⁷⁾ Electrodeposited gold-copper alloys have a hardness of up to 450 Knoop after heat treatment, but these would possibly be precluded in an oral environment because of their high copper content (25-30 per cent). Bright acid golds deposited from solutions of low free cyanide content have been developed with hardness values from 120 to 300 Knoop.⁽⁶⁾⁽⁷⁾ Metals such as cobalt, nickel and iridium are co-deposited with the gold but the gold content is not less than 92 per cent (22 ct.).

The thickness of gold required clinically will have to be assessed. A thickness of up to 0.0005 inch when applied to castings does not appear to have any effect on their fit. For comparison, jewellery is labelled heavy gold electroplate when articles have a thickness of gold greater than 0.0001 inch.⁽⁸⁾

Warning: When cyanide solutions are used certain precautions must be observed. The nickel electrolyte is acid and must not come into direct contact with the gold electrolyte. The castings must be rinsed in water when transferred from one bath to the other.

To remove any possible traces of residual cyanide after gold deposition the casting is washed thoroughly in alternate hot and cold water rinses and placed in 10 per cent hydrochloric acid for several hours.

Cyanide free electrolytes that could be of interest to the dental profession have been developed. Their deposits are not as hard as bright acid gold deposits and details of their properties have not yet been made fully available.*

Summary

A method suitable for gold-plating cobalt-chromium castings, and other chrome-containing alloys by surface activation and nickel deposition has been described. This process has the advantage of gilding clasps or an entire casting with a hard bright gold.

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Die Quintessenz der Arbeit:

Vergolden von Chrom-Kobalt-Legierungen

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Aus: Australian Dental Journal 14, Nr. 2, 79—81 (April 1969)

(Gold-plating of cobalt-chromium)

Mit der Einführung von Chrom-Kobalt-Legierungen in die Zahnheilkunde eröffneten sich völlig neue Möglichkeiten für die Konstruktion herausnehmbarer Teilprothesen. Trotz aller unbestrittenen Vorteile dieser Metalle (z. B. Härte, geringes Gewicht, Korrosionsfestigkeit) erscheint ihr silbernes Aussehen manchem Patienten als nicht annehmbar und störend, so daß häufig der Wunsch vorgetragen wird, das ganze Stück (oder zumindest die sichtbaren Klammern) sollten vergoldet sein.

Da Chrom nun einen passivierenden Effekt auf Oberflächen hat — es bildet eine beständige, schützende Oxidschicht aus, die das Metall gegen Korrosion schützt — kann eine solche Prothese nicht ohne weitere Vorbehandlung vergoldet werden.

Um ein günstiges Verfahren hierfür zu erhalten, wurden verschiedene Versuche durchgeführt. Hierbei erwiesen sich einige Elektroverfahren, die zur Oberflächenaktivierung von Stahl angegeben werden, als ungeeignet. Als Elektrolyt für ein erfolgreiches Vorgehen wird eine Lösung benützt, die von WOOD angegeben wurde, und aus 240 g Nickelchlorid, 86 ml 10-n Salz-

säure besteht und mit destilliertem Wasser auf 1 Liter aufgefüllt wird.

Das zu vergoldende Stück wird an einem Kupferdraht in die Lösung gebracht. Die Behandlung wird bei 21 bis 26° C durchgeführt und soll anodisch 2 min bei einer Stromstärke von 3,2 amp/dm², und anschließend mit vertauschten Elektroden (das Gußstück ist jetzt die Kathode) bei gleicher Stromstärke für 6 Minuten erfolgen. Nach dieser Vorbehandlung wird das Gußstück gründlich gespült und sofort anschließend mit einer der üblichen Methoden vergoldet.

Metallographische Photoaufnahmen von derart vergoldeten Gußstücken zeigen deutlich, daß die Chrom-Kobalt-Legierung genügend gereinigt und angegriffen wird, um sowohl einen guten chemischen wie auch — durch Bildung einer unregelmäßigen, rauhen Oberfläche — mechanischen Kontakt zwischen dem Guß und dem Gold zu gewährleisten. Dieses Verfahren wird daher anstelle des bisher üblichen bloßen Aufschwemmens mit Goldlot empfohlen, da erstens der Kontakt der beiden Metalle wesentlich fester ist und zweitens eine gleichmäßige Beschichtung erfolgt. □

Electroformed metal palates for complete dentures

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The explosive or high energy forming of stainless steel for palates in complete dentures¹⁻³ has revitalized the profession's interest in the use of metal as a denture base material.

Over a long period of dental history much thought has been devoted to swaging and casting techniques to produce metal palates. The manipulative skill required to manufacture them by swaging—the use of 2 dies and 4 or 5 counter-dies to produce a swaged palate (in aluminium, silver, platinum, iridio-platinum, palladium-silver, stainless steel or gold alloys⁴⁻⁷—emphasizes the value placed on the advantages of a metal palate.

The casting of dental bases in tin was reported in 1820.⁴ Since then, three basic metals (tin, aluminium, gold) alloyed with other metals (such as silver, copper, bismuth, nickel, magnesium, platinum and palladium) have been tried and gradually discarded.^{4, 6, 7} Today cast palates are usually constructed of cobalt-chromium alloys.

Considering the time and effort spent in both the swaging and casting techniques to produce a metal palate, there is a definite interest, if not a need, for the metal palate as a supplement to denture construction.

The advantages of a metal palate, as given by Swenson⁸ are: (1) better thermal conductivity as compared to the resins, (2) increase in tissue tolerance, because of a less irritating surface, and increase in stimulation from heat and cold, and (3) reduction of bulk across the palate which is an important factor to the patient, as it creates more tongue space.

The disadvantages of a metal palate are cost and maintenance problems, such as rebasing and posterior palatal seal adjustment.⁹ The casting of a thin, large denture base with a non-precious metal, such as the cobalt-chromium alloys, is relatively expensive due to the intricate technique involved. The degree of fit is questionable and adjustments are difficult, if not impossible.

STAINLESS STEEL

The explosive forming of stainless steel provides a simpler technique but, as with the other methods in use, it still has the disadvantage that each palate is prepared on an individual basis during manufacture. This is time-consuming and

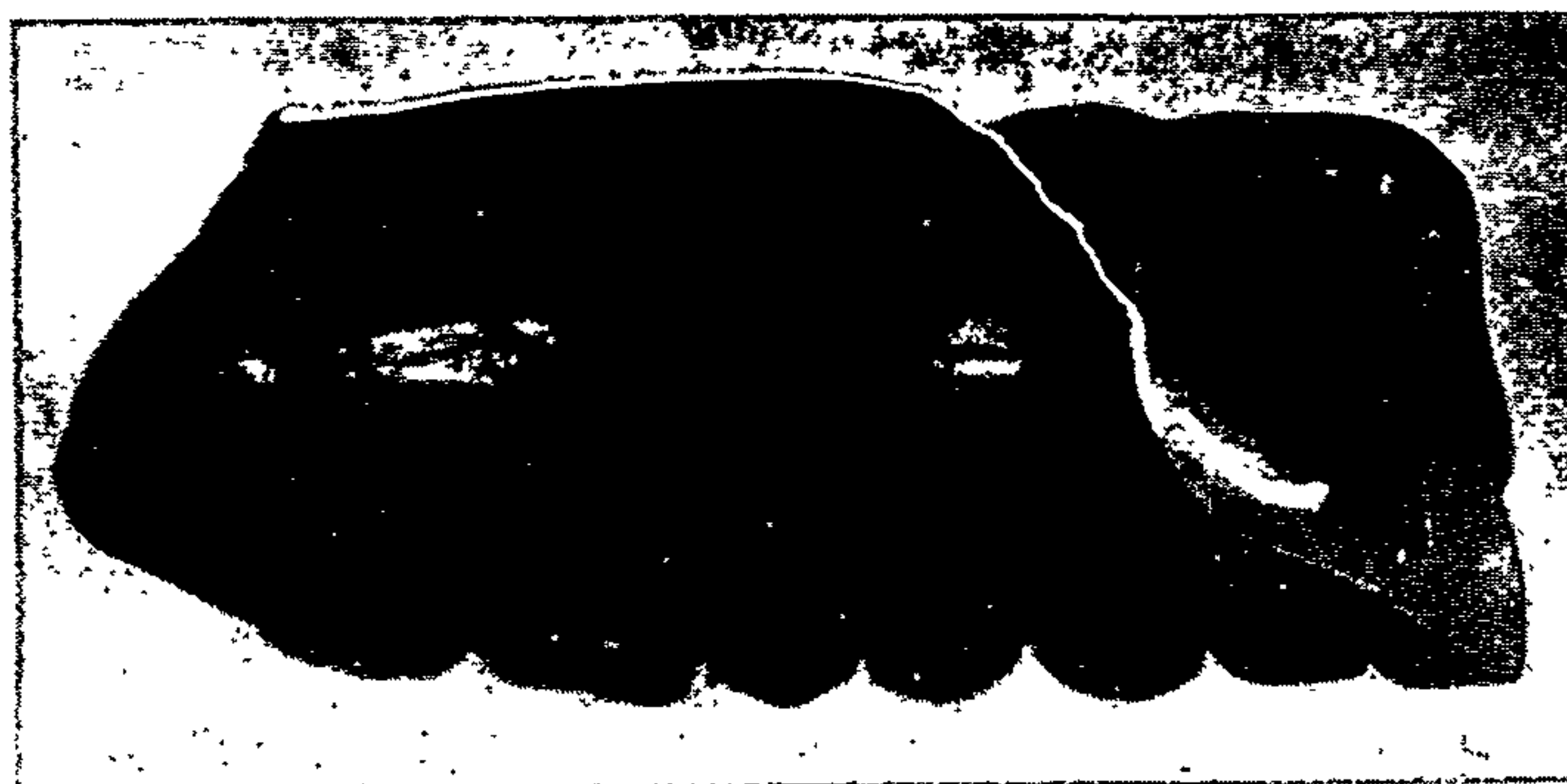


Fig. 1. *A* Anteroposterior cross-section of a denture with an electroformed metal palate. Note the increased thickness of metal embedded in the acrylic resin. Heavier electrodeposition of metal is used in this area to give the palate greater strength in its retentive area.

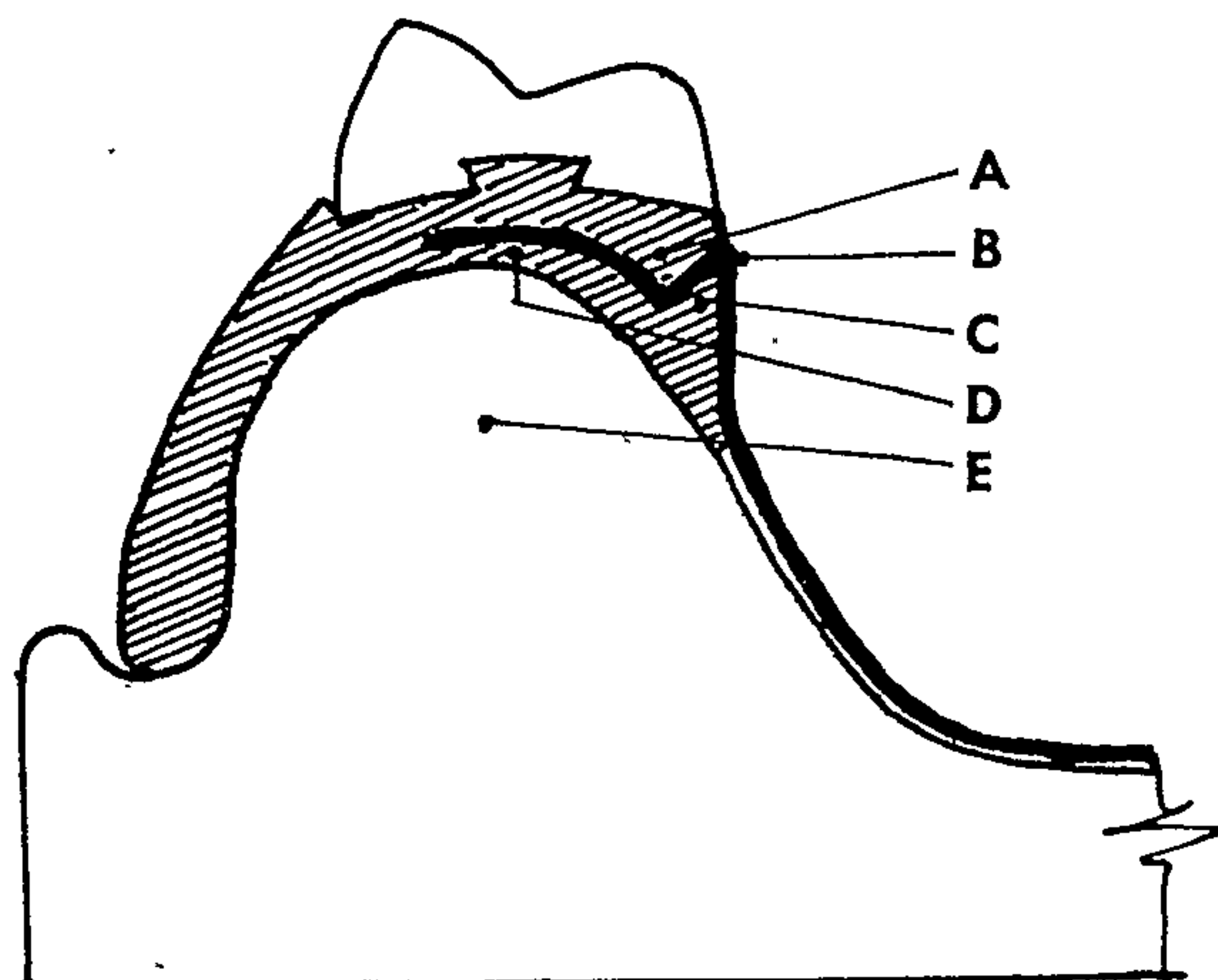


Fig. 1. *B* Diagrammatic cross-section of posterior region shows how an "acrylic sandwich" is formed around the metal. The finishing line provides for bulk of acrylic on both the lingual surface (*A*) and the tissue surface (*C*). (*A*) lingual finishing line, (*B*) metal electroform, (*C*) palatal finishing line, (*D*) relief area on crest of ridge, and (*E*) stone cast.

a method of multiple production would be an advantage if a large number of palates could be produced concurrently and with a minimum of supervision. With this in mind, a technique was devised to electroform metal palates on artificial stone casts. A low stress nickel was used for electroforming and, to prevent tarnish in the oral environment, the nickel was electroplated with gold and in some cases with rhodium.

METHOD

Modification of master cast and duplication before electroforming procedures. This is necessary to produce: (a) a retentive area for embedding the metal palate



Fig. 2. A Half thickness of wax placed on the crest of the ridge of the cast to be duplicated.

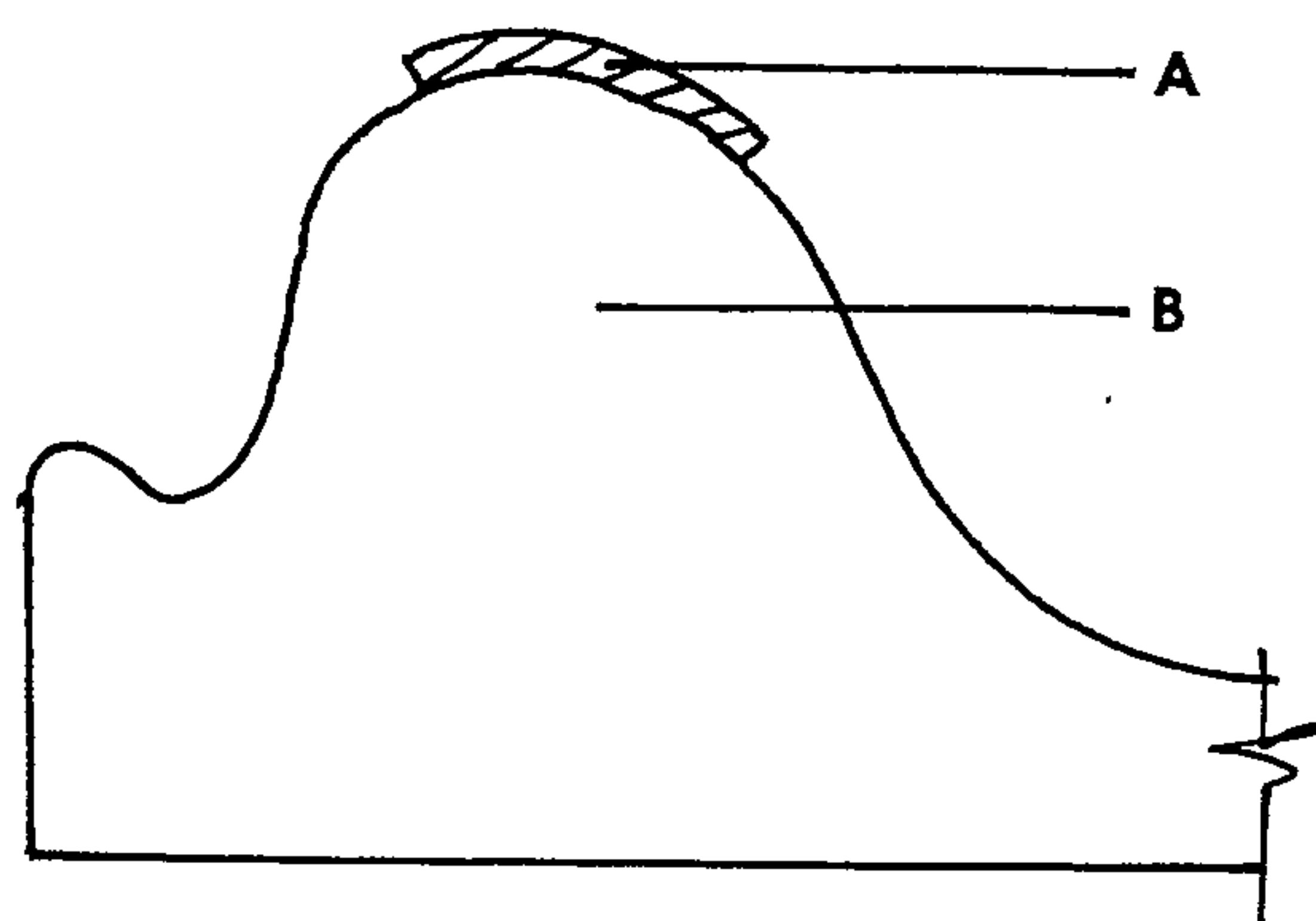


Fig. 2. B Cross-section diagram of the design. (A) wax relief, and (B) stone cast.

into the denture and (b) a finishing line for the acrylic resin on both sides of the metal palate (Fig. 1).

1. Place a half thickness of sheet wax around the crest of the ridge on the original cast (Fig. 2).

2. Position a preform (as used for casting the finishing line on cobalt-chromium dentures) over the half thickness of sheet wax (Fig. 3).

3. Duplicate the prepared original cast in the usual manner and pour in artificial stone (Fig. 4).

4. Trim the duplicated cast.

5. Drill 2 small holes into the base of the cast about 0.5 cm. deep.

6. Bend the wire contacts as shown in Fig. 4 and cement the wire into the prepared holes with zinc phosphate cement (Fig. 5).



Fig. 3. *A* The preform is added to the wax relief. The cast is ready for duplication.

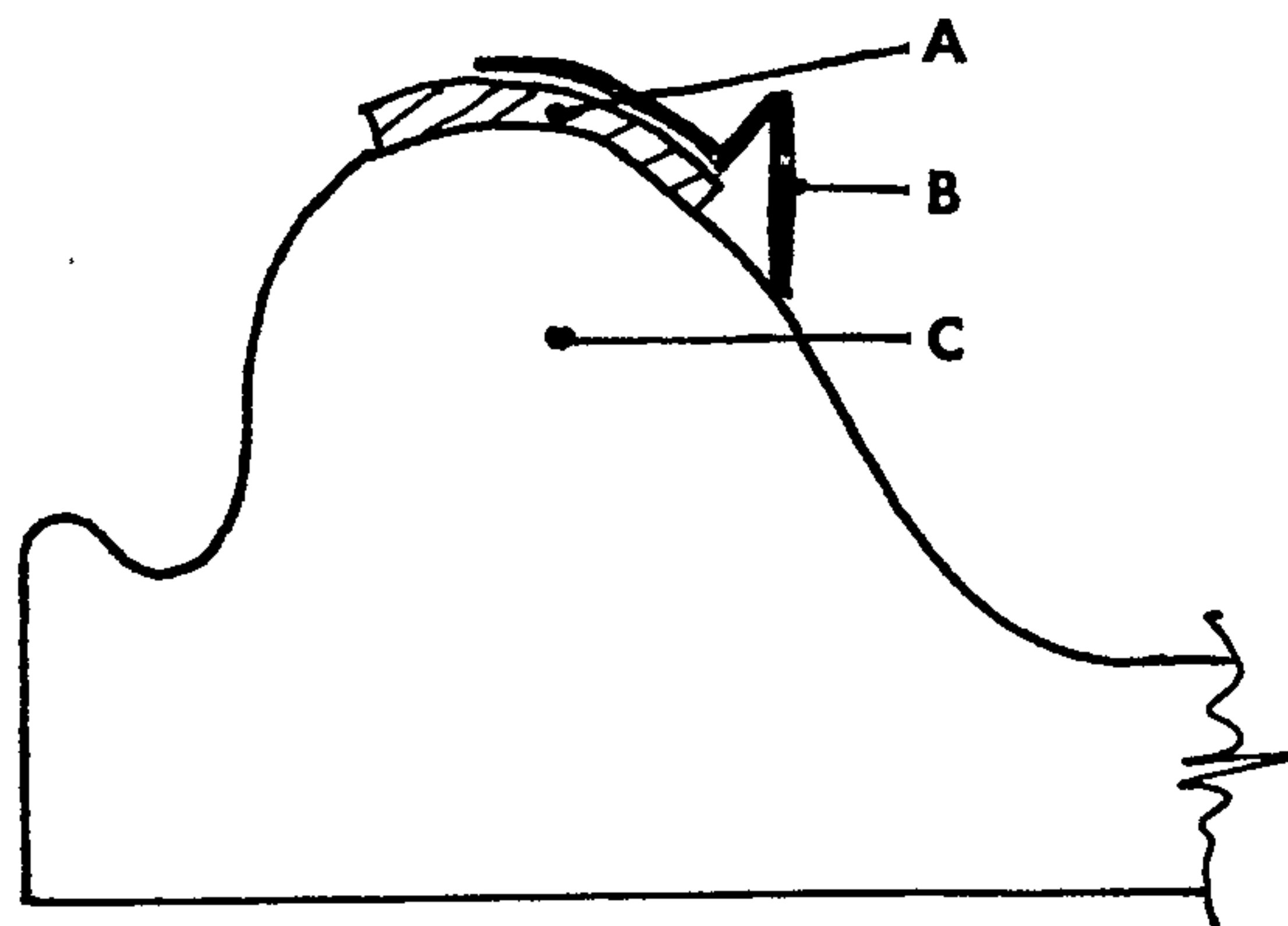


Fig. 3. *B* Cross-section diagram of the design. (*A*) wax relief, (*B*) preform, and (*C*) stone cast.

METALLIZING THE DUPLICATE CAST

Preparation of cast with conductive silver paint.*

1. Paint a thin layer of the conductive silver paint onto the area of the cast to be electroformed.
2. Repaint over the conductive silver paint with solvent ether.
3. Paint the wires that are inserted into the cast. This ensures that the current will be conducted to the areas to be electroformed (Fig. 5).
4. Paint the untreated parts of the stone cast with an epoxy resin to protect the exposed areas from chemical attack by the electrolyte.

*Degussa Conductive Silver 200, Degussa-Frankfurt au Main.

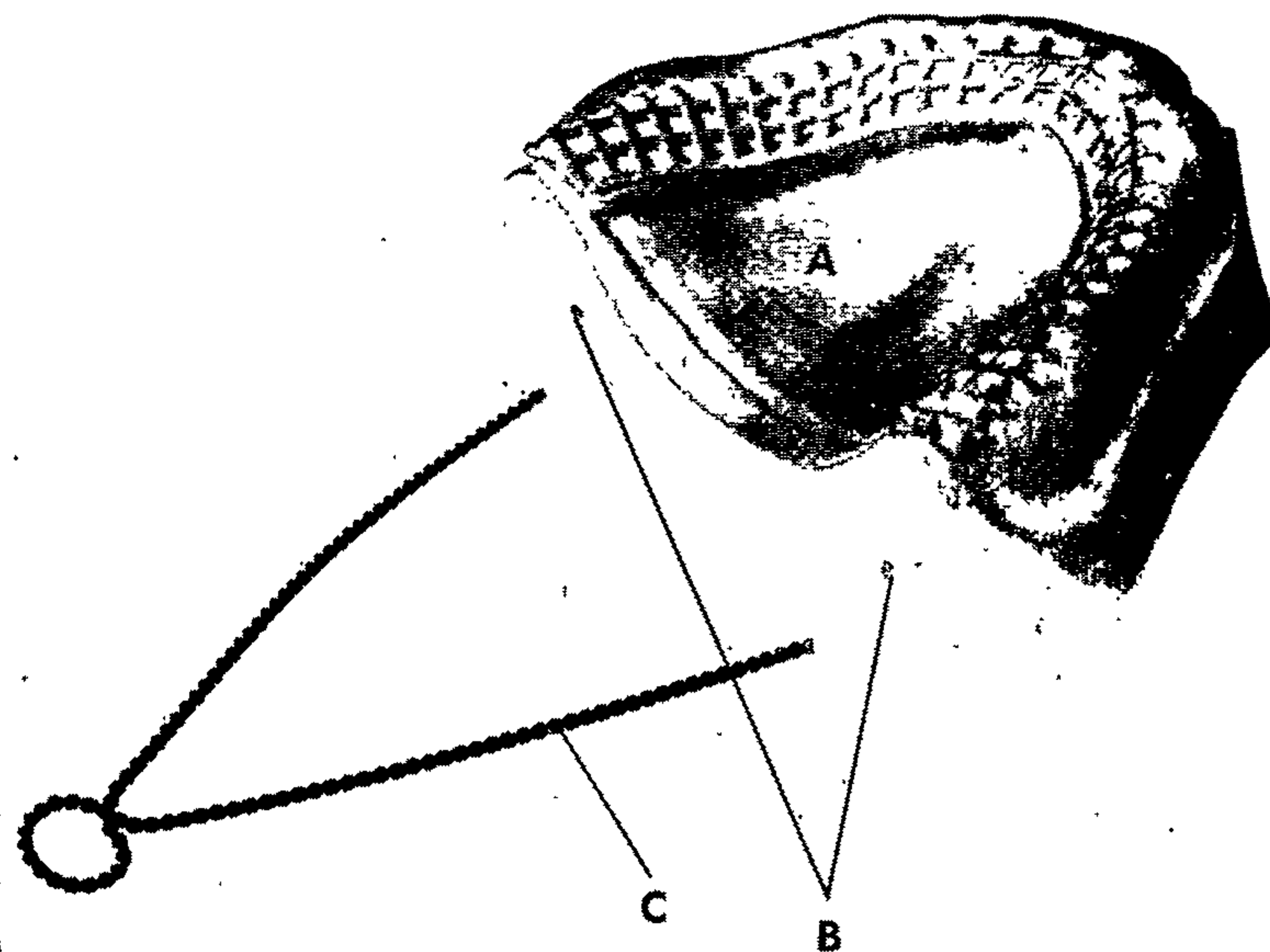


Fig. 4. Artificial stone duplicate cast (A) with holes (B) drilled in the base of the cast for insertion of the wire contact (C).

ELECTROFORMING

1. Place 8 liters of electrolyte solution (Barrett's¹⁰ modified solution)* in a P.V.C.† tank with thermostatic control.
2. Maintain a temperature between 100° F. and 120° F. (38° C. and 49° C.).
3. Maintain cathode current densities between 20 and 60 amp. per square foot (2.2 and 6.6 amp. per square decimeter). 23 palates were produced in this manner (Fig. 6).
4. Electroform for 16 to 18 hours to produce a palate rim thickness between 0.017 and 0.045 in. and a palate thickness of 0.004 to 0.009 in.

FINISHING

1. Wash and dry the electroformed cast (Fig. 6).
2. Cut out the palatal outline required with separating discs (Fig. 7).
3. Lift the palatal section from the stone cast (Fig. 8).
4. Polish in usual manner. Polishing is generally not required before gold or rhodium plating.
5. Gold or rhodium plate the electroformed palate.
6. Use bright acid-gold deposits because of the increased hardness values (300 D. P. N.) and high gold content (92 per cent).^{11, 12}
7. Replace the palate on the original cast.

*Nickel sulfamate 450 g/l, Nickel Chloride 30 g/l, Boric Acid 45 g/l, pH 3.5-5.0.

†A. C. I. Plastics.

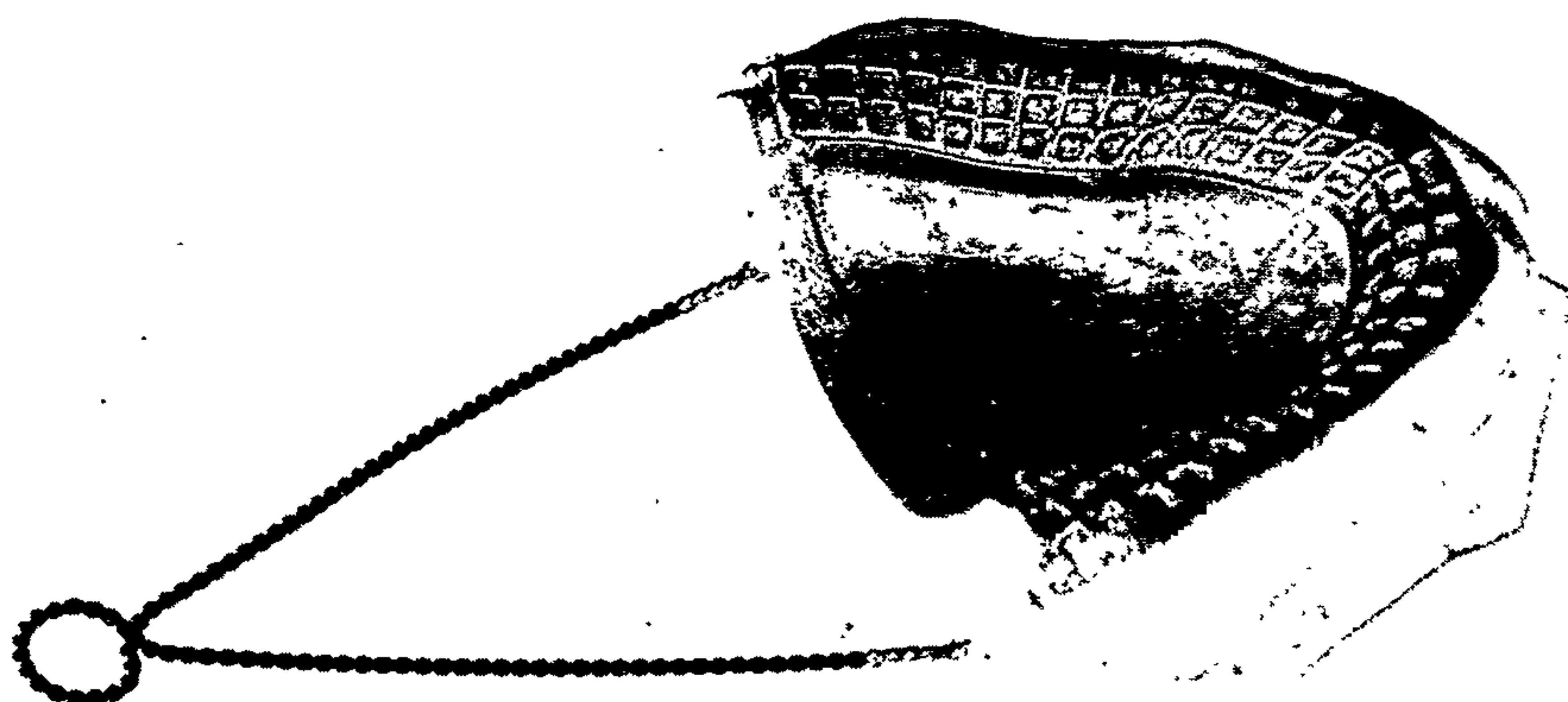


Fig. 5. The wire contact is cemented in place and the cast is metallized to provide a conductive surface from the contact wires to the area to be electroformed.

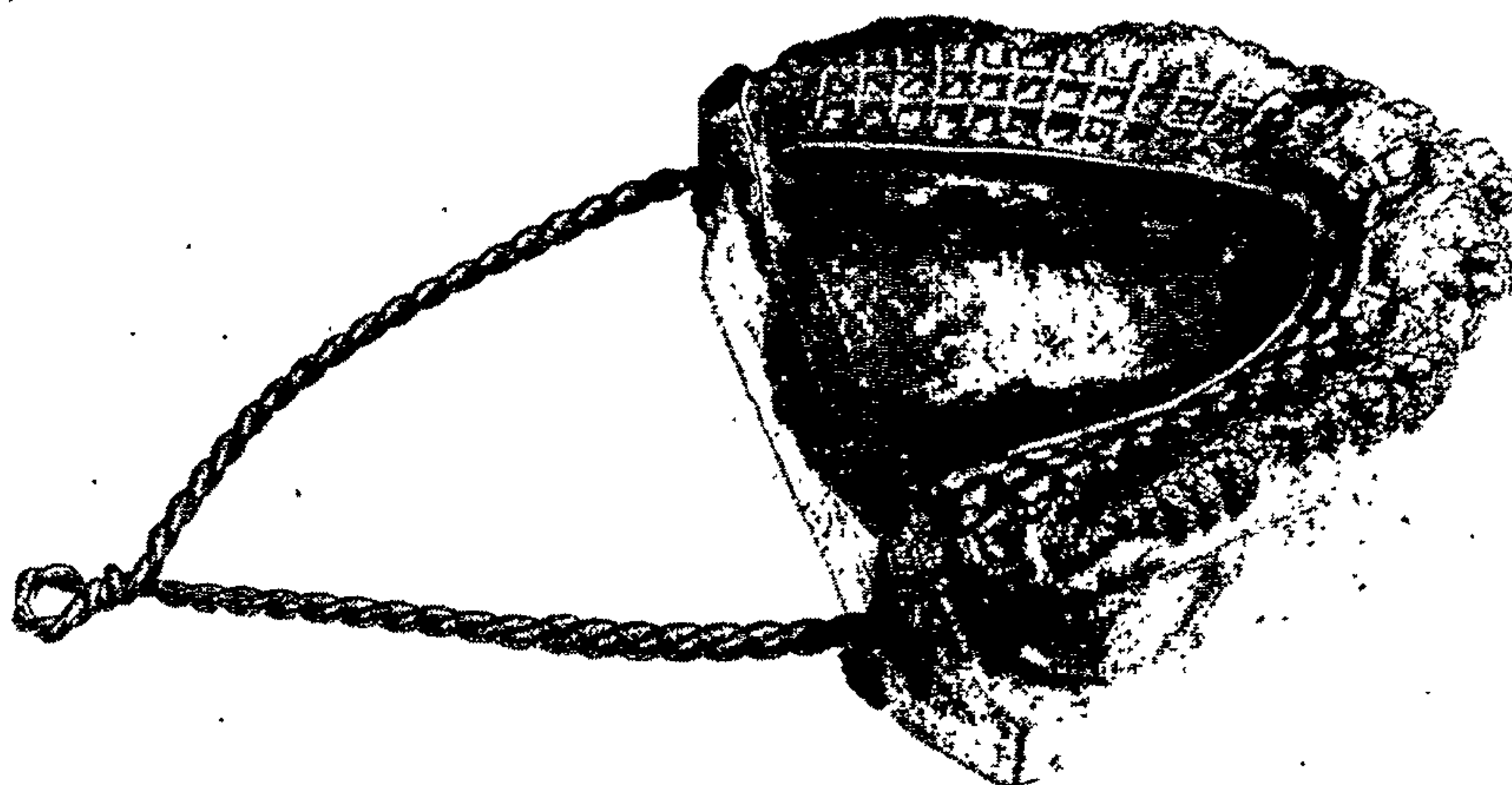


Fig. 6. The cast after electroforming with a low-stress nickel. The nodular boundary is due to current concentration at the periphery of the electroform.

8. Set the teeth and wax the denture to the palate.
9. Invest the wax-up and process and finish the denture in the normal fashion (Figs. 9 and 10).

DISCUSSION

Metallizing is a method of providing a thin continuous metal surface on the model (cast) so that it will conduct electricity.

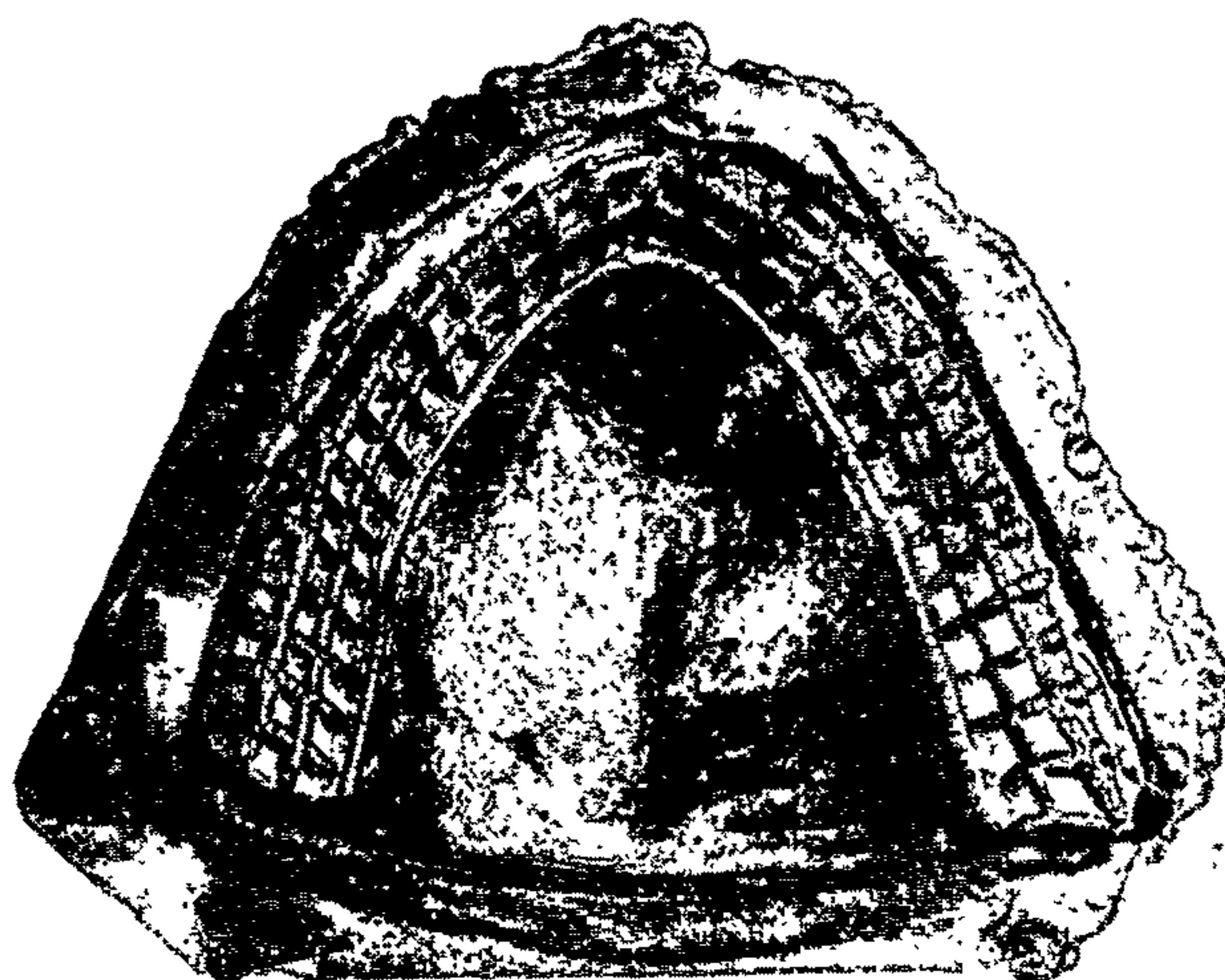


Fig. 7. The electroformed palate is enscribed with a separating disc to the required shape.

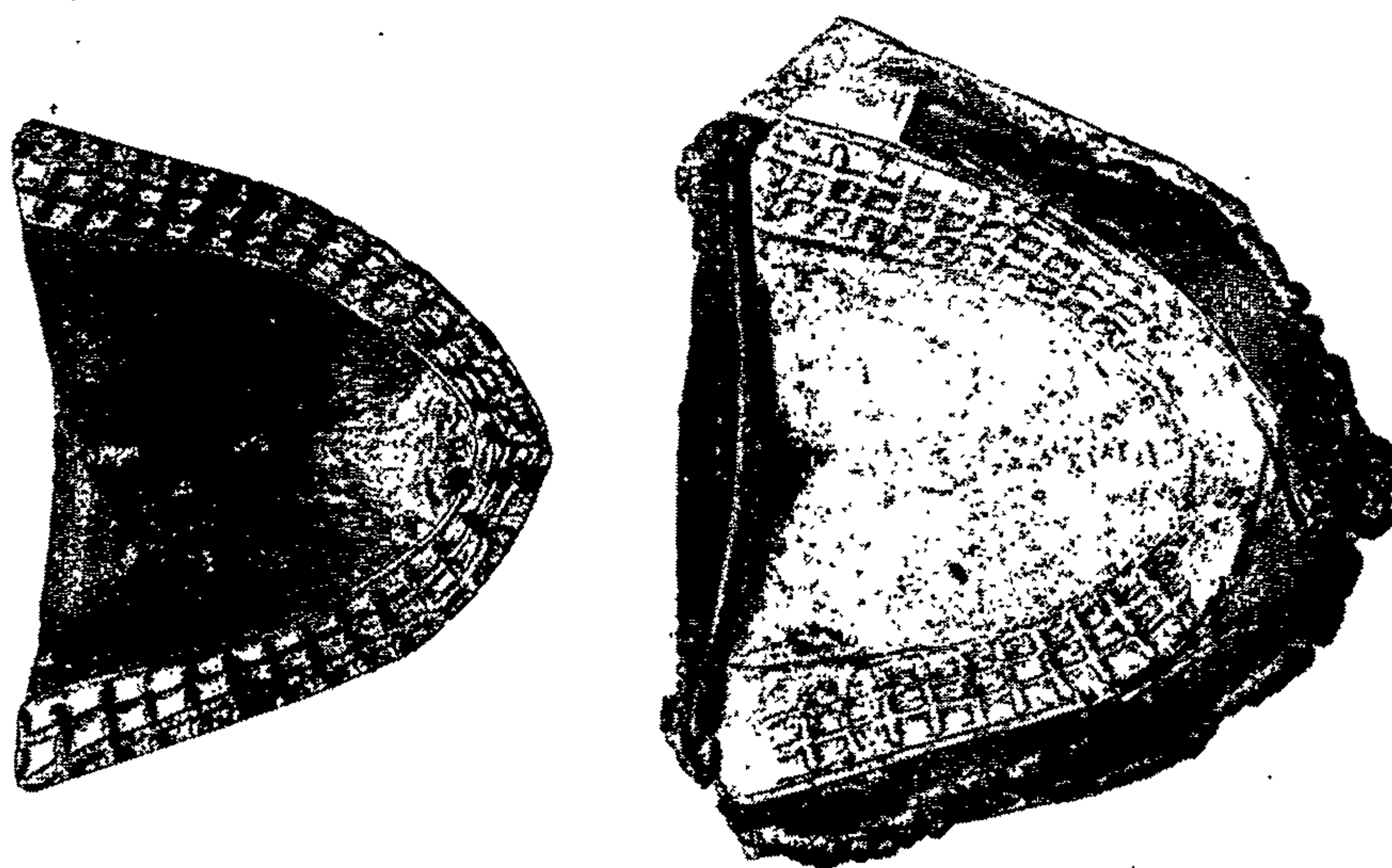


Fig. 8. Electroformed palate removed from the artificial stone cast. After receiving a protective coat of gold or rhodium, it is ready to be inserted in the complete upper denture.

A silver reduction method¹³ and an electroless copper reduction method* were tried. For these processes it is necessary to make the cast non-absorptive. This is done by boiling in wax, or treating with epoxyresin, and in the case of electroless copper,

*Enplate cu-400 process.

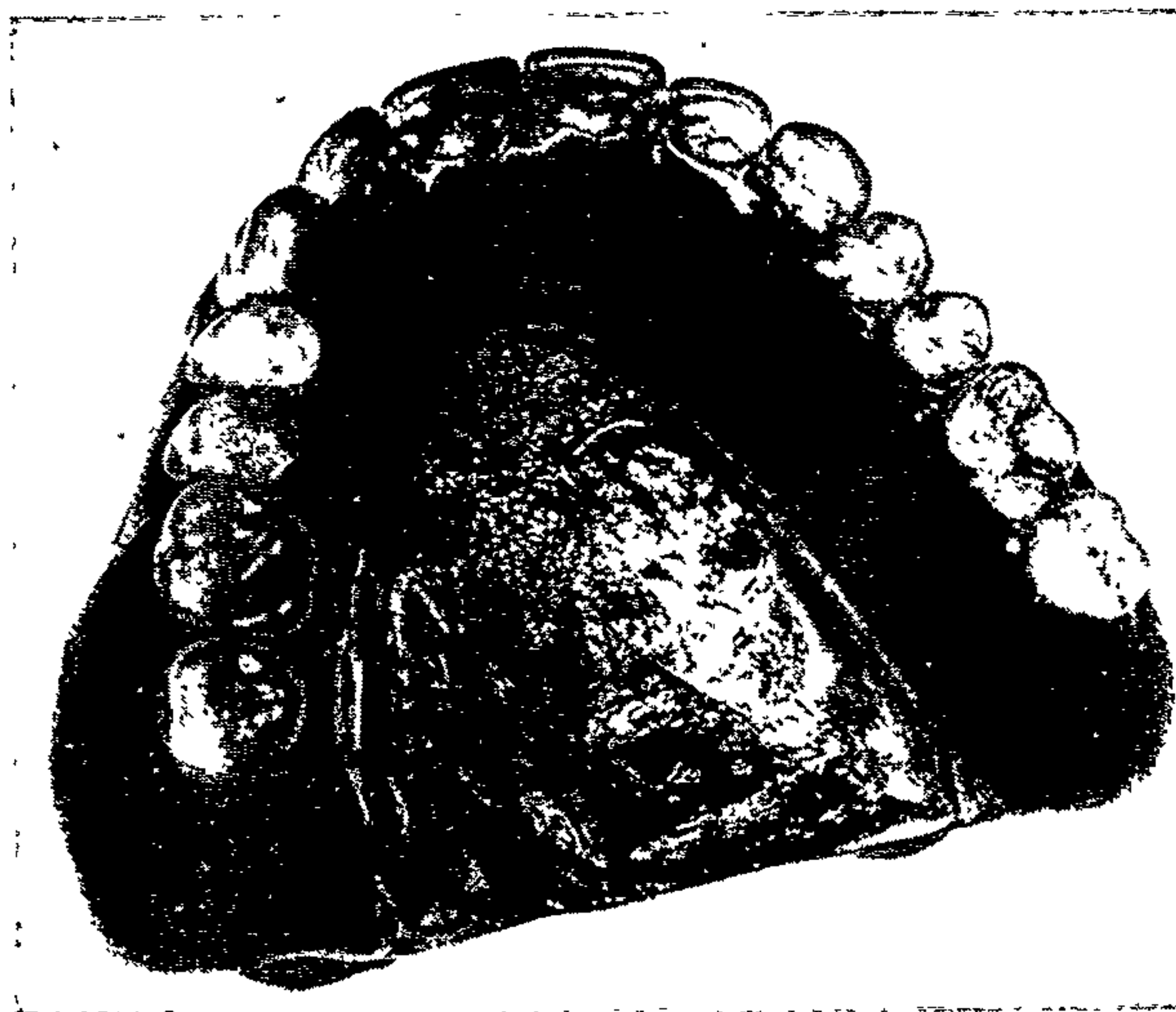


Fig. 9. A complete denture with an electroformed metal palate. Note the finishing line between the metal and the acrylic resin.

by treatment with A.B.S. (acrylonitrile-butadiene-styrene) polymer dissolved in methyl ethyl ketone.

It was considered that these procedures could lead to a discrepancy in the fit of the electroformed palate, and, as the chemicals tended to stain the hands, other avenues were considered.

Silver oxide paints with oil* and water† bases were considered. These paints are reduced to metallic silver by the application of heat but the required temperatures often destroy the cast by the formation of steam which blisters the conductive surface. However, when the cast is dried in an oven after painting and the surface reduction is initiated with the flame from a compound torch, a good surface is produced.

The simplest and most effective method is to use a conductive silver paint‡ to cover the area that requires electroforming. This paint must be in contact with the wires that are inserted so that the current is conducted to the area to be electroformed (Fig. 5). Because the resistance of a conductor varies inversely with the cross sectional area (the thinner the conductor, the greater its resistance) and is directly proportional to its length (the longer the conductor, the greater its resistance), the contact area at the wires is made as large as possible. The manufacturer recommends heat treatment with the conductive silver to improve its conductivity. By using solvent ether in conjunction with the paint, the spreading factor is increased, the thickness which could affect the fit is decreased, and the conductivity is greatly improved, so that the heat treatment is unnecessary. This

*Degussa Silver Mixture 112 (oil) Degussa-Frankfurt au Main.

†Degussa Silver Mixture C (water base) Degussa-Frankfurt au Main.

‡Degussa Conductive Silver 200 Degussa-Frankfurt au Main.

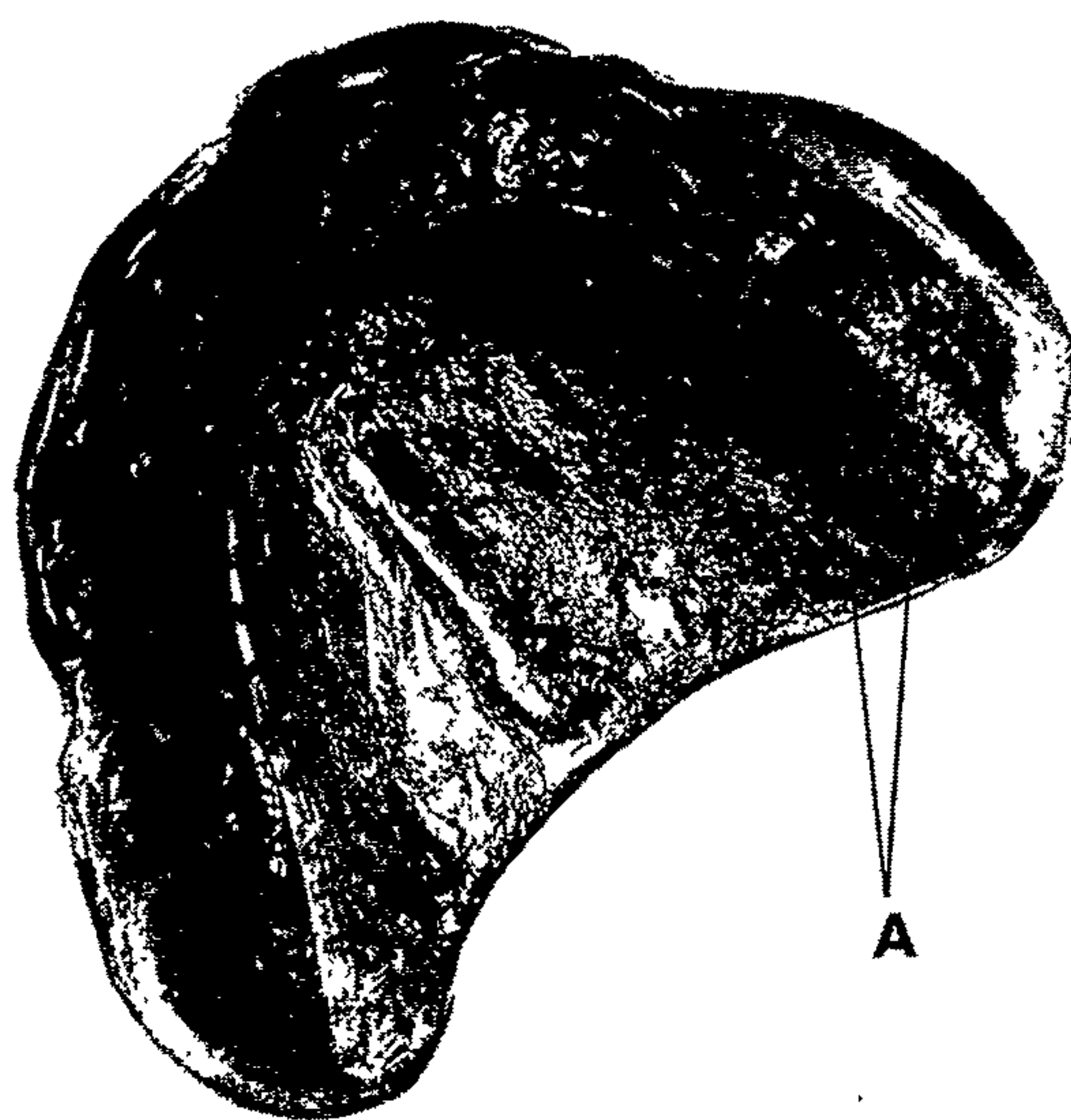


Fig. 10. The basal surface of a complete upper denture with an electroformed metal palate. Note the finishing line between the metal and the acrylic resin. Holes for additional retention have been placed in the embedded metal (A). This procedure is optional.

solvent removes the etheric oils and organic components that are used as binders and produces a closer contact between the particles of silver in the paint.

The use of nickel electroforming as an industrial production method is rapidly growing because electroforming can: (1) produce intricate shapes, (2) reproduce with great fidelity close dimensions and varied surface textures, (3) achieve fine detail, and (4) be adapted to large scale production. Some of the established uses are the building up of badly worn or over machined surfaces of engineering components,¹⁴ electrotyping, the manufacture of dies for moulding plastics, and the production of various components such as radar wave guides, surface roughness standards, and stampers in the phonograph industry.¹³

Nickel has a relative density of 8.9 Gm. per c.c. which is comparable to that of cobalt-chromium and stainless steel. Depending on the electrolyte used and the operating conditions, nickel has an ultimate tensile strength of 55 to 110×10^3 p.s.i., a hardness range of 130 to 275 D.P.N. and a percentage elongation of 32 to 5 per cent.¹³⁻¹⁵

The relationship of tensile strength, hardness, and ductility with nickel electrodeposits is interrelated. As the tensile strength increases so does the hardness, while the ductility or percentage elongation decreases.¹⁵

Additionally, there are internal stresses which bear no relationship to the above properties. These forces can be of sufficient magnitude to either distort or cause separation of the electroformed metal from the cast. Electrodeposited metals can have a tensile or contractive stress that will crack, flake, or bend the deposited metal, or a compressive or expansive stress that causes blistering of the deposit or buckling of the electroform.

Deposits from the Watts type electrolyte^{13, 15, 16} have a tensile stress of 18,000 to 23,000 p.s.i. The deposits from the fluoborate electrolyte^{13, 15} have a tensile stress of 15,000 to 24,000 p.s.i. These stresses are undesirable but they are not so critical if the electrodeposited metal is permanently attached to a basis metal. With electroforms, the tensile stress should not exceed 10,000 p.s.i.¹³ to ensure accurate reproduction. The internal tensile stress of the sulfamate bath is between 1,000 and 6,000 p.s.i.¹³ Kendrick¹⁶ reported that a concentrated nickel sulfamate bath (600 gm. per liter) operated at a current density of 50 amp. per square foot (5.5 amp. per square decimeter) gave an internal compressive stress of 14,000 p.s.i. in the deposit. At a current density of 200 amp. per square foot (22 amp. per square decimeter) the deposit was stress-free.

The sulfamate bath can be operated over a wide range of conditions. At a temperature of 25° C. the maximum current density is 140 amp. per square foot (15 amp. per square decimeter) and at 75° C. the maximum current density is increased to 300 amp. per square foot (32 amp. per square decimeter).¹⁷ This would give a corresponding rate of deposition of 0.007 inch per hour and 0.015 inch per hour respectively. However, higher current densities give greater inequalities of thickness over the cathode area, and higher temperatures introduce the coefficient of expansion as a factor requiring compensation.

Kendrick¹⁶ produced a stress-free nickel deposited from a concentrated sulfamate solution (600 gm. per liter) at a current density of 200 amp. per square foot (22 amp. per square decimeter) and a temperature of 60° C. The rate of deposition was 0.01 inch per hour.

The maximum rate of deposition with the solution as described in this article was 0.0025 inch per hour. This could have been increased with improved operating conditions.

In some of the experiments the region of the posterior palatal seal was built up to a greater thickness than the general palatal area by the use of contoured anodes. These palates were more rigid.

The nickel deposited on the crest of the ridge was 4 to 5 times as thick as that in the palatal area (Fig. 1A). This gave the palate greater strength where it was embedded in the acrylic resin without reducing the tongue space.

A series of "laminated" palates formed by alternate layers of nickel and copper were produced. The use of composite metal coats, although an interesting speculation, only offers greater technical difficulties.

Electroforming can be done in the dental laboratory but it is more suited for use on a production basis as a great number of palates can be produced at the same time if adequate equipment is installed.

CONCLUSION

A new method for producing metal palates for complete upper dentures by electroforming is described. Electroforming provides greater accuracy of reproduction than casting or swaging techniques. There are fewer steps and materials involved, and the electrodeposited metal is not subject to casting discrepancies of the metal itself, or of dies and counter-dies.

I would like to thank B. W. Armstrong, Senior Lecturer, School of Metallurgy, University of New South Wales, and A. R. Docking, Director of The Commonwealth Bureau of Dental Standards, for their advice and assistance in the preparation of this article.

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The quintessence of the article:

Electroformed metal palates for complete dentures

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Address: 70 Gynea Rd., Sydney, Australia

From: Journal of Prosthetic Dentistry 23(2):207—216

A great deal of professional interest exists everywhere in finding an improved method for constructing the palatal portion of an upper complete denture of metal (Fig. 1).

Historically, the endless search for casting procedures began in 1820 with the use of cast tin for baseplates. Since then alloys of three basic metals, tin, aluminum and gold with silver, copper, bismuth, nickel, magnesium, platinum and palladium have followed. Today palatal plates are usually constructed of chromium-cobalt alloys.

The advantages of a metal palate are:

1. better thermal conductivity as compared to resins,
2. better tissue tolerance because of a less irritating surface, and an increase in stimulation from heat and cold,
3. reduction of the size of the plate, giving the tongue more space for movement.

The disadvantages of the metal palate are cost and maintenance problems. The casting of a thin denture base from a non-precious metal such as the chromium-cobalt alloys is very costly because of the technique employed. Adjustments are difficult, and almost impossible when the degree of fit is questionable.

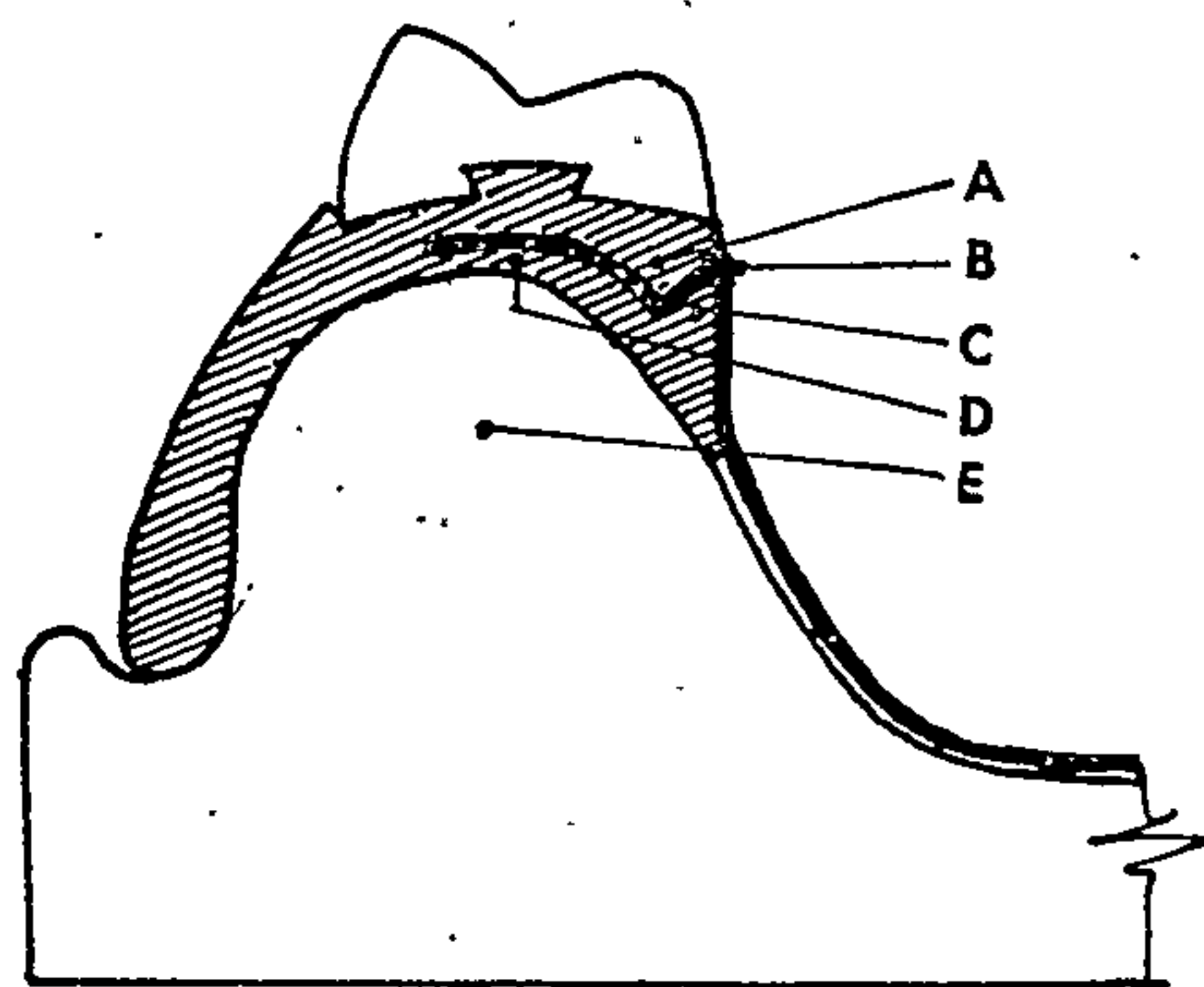


Fig. 1

A cross-section shows how the metal plate is incorporated in the acrylic resin. The angled form of the plate provides for bulk of acrylic on the lingual and the palatal surface. (A) lingual finishing line, (B) metal plate, (C) palatal finishing line, (D) relief area on crest of ridge, (E) stone cast

A method will be described here which involves the construction of palatal plates using electroforming.

Technique

The maxillary impression is prepared for plating and then constructed in a modified form from a special stone cement. Added to this are the relief areas on the crest of the ridge for finishing with acrylic resin, using a strip of wax and a prepared retention net. Two

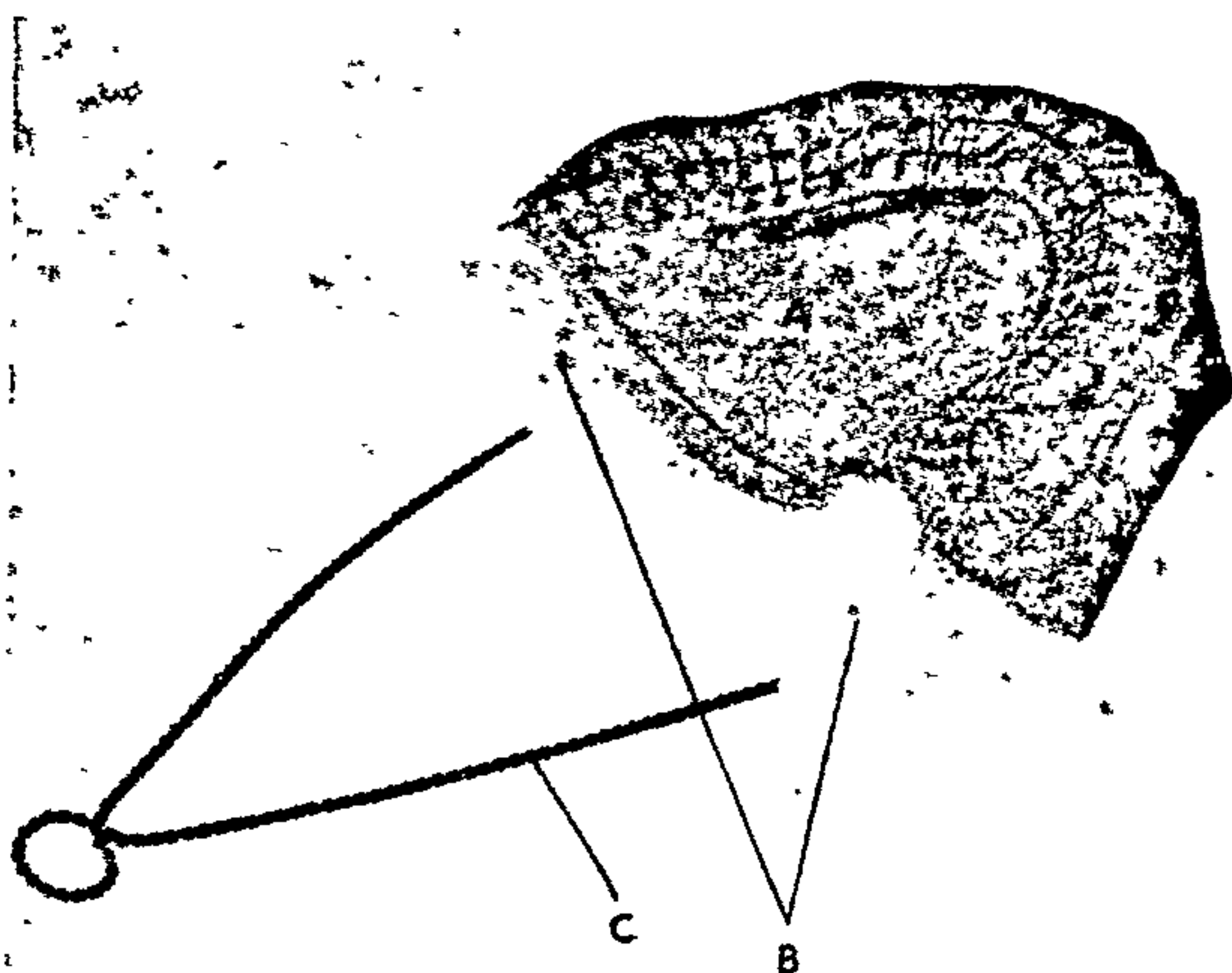


Fig. 2

A wire loop (C) is inserted in two small holes (B) of the modified model (A); on the crest of the ridge of the acrylic relief area

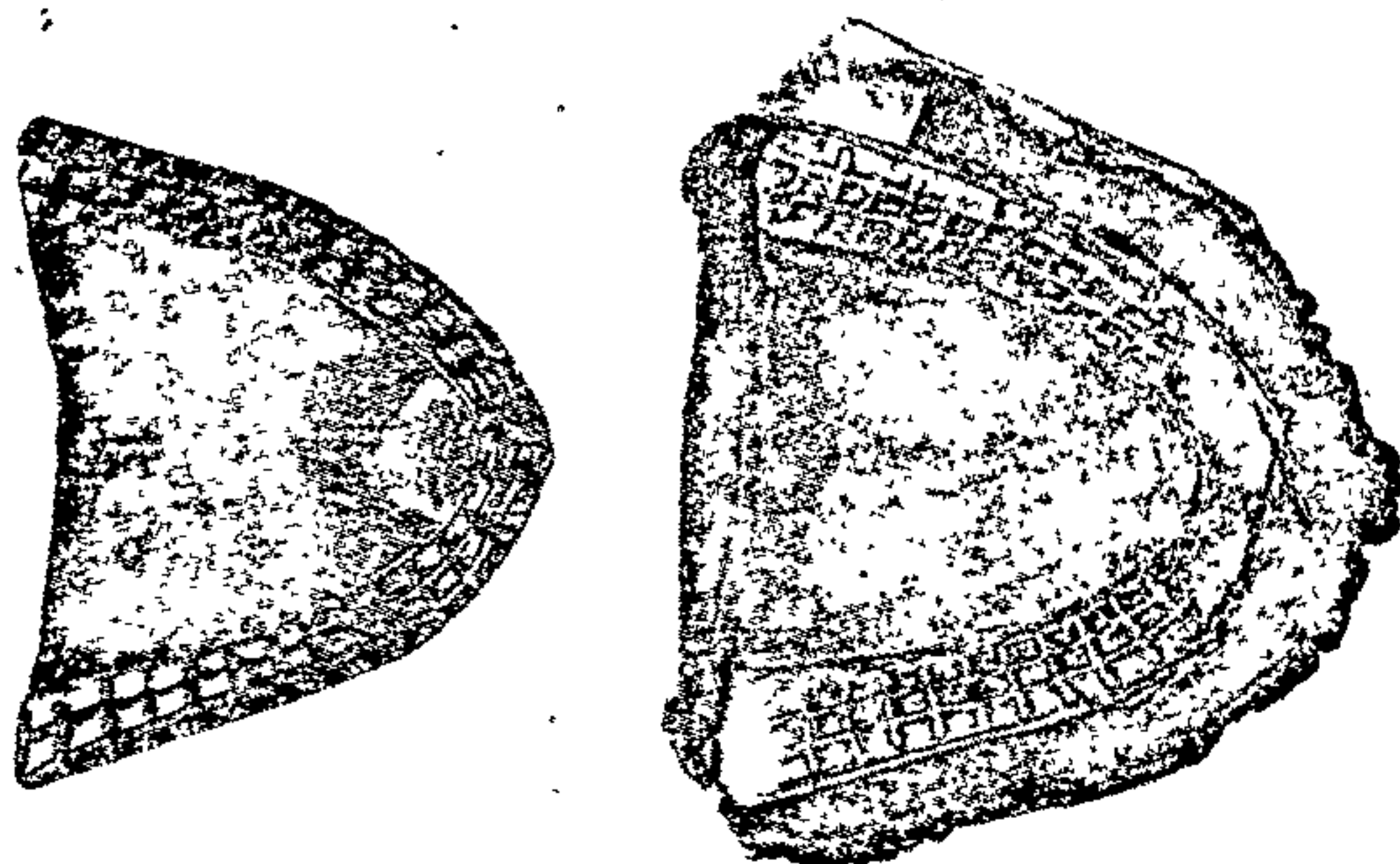


Fig. 3

Electroformed plate (right) removed from the model (left)

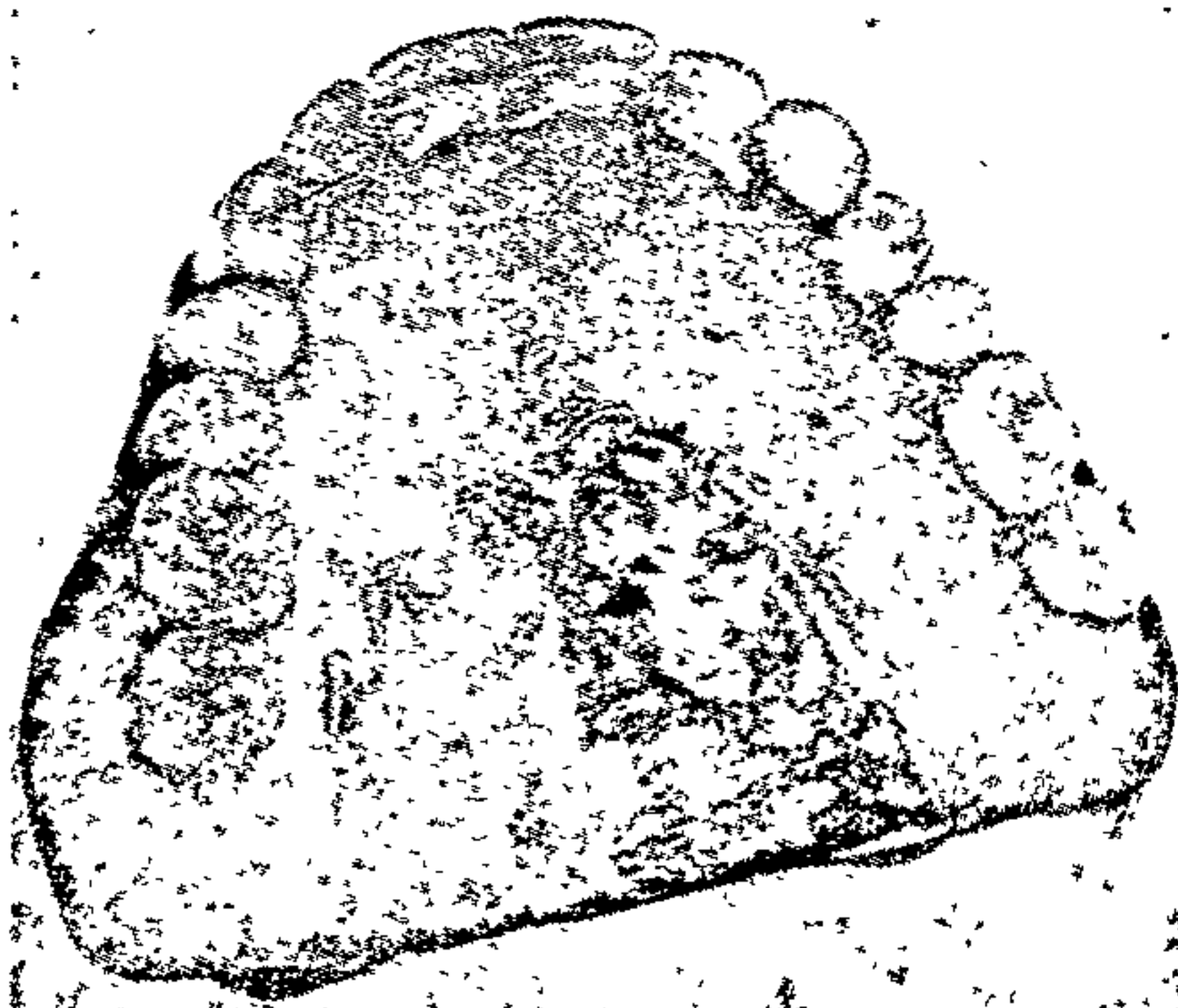


Fig. 4

The finished complete upper denture with metal palate

small holes are bored into the base of the cast about 5 mm deep. The wire needed for electric current is cemented into these holes (Fig. 2).

The portions of the model to be plated are painted with a thin layer of silver paint (Degussa 200). The wires are similarly painted. The rest of the model is protected with a coating of epoxy resin varnish.

Plating is done in an 8 liter tank containing electrolyte solution at a temperature of 38 to 49° C. The cathode current should be between 220 and 660 amp./cm². In 16 to 18 hours a palate rim

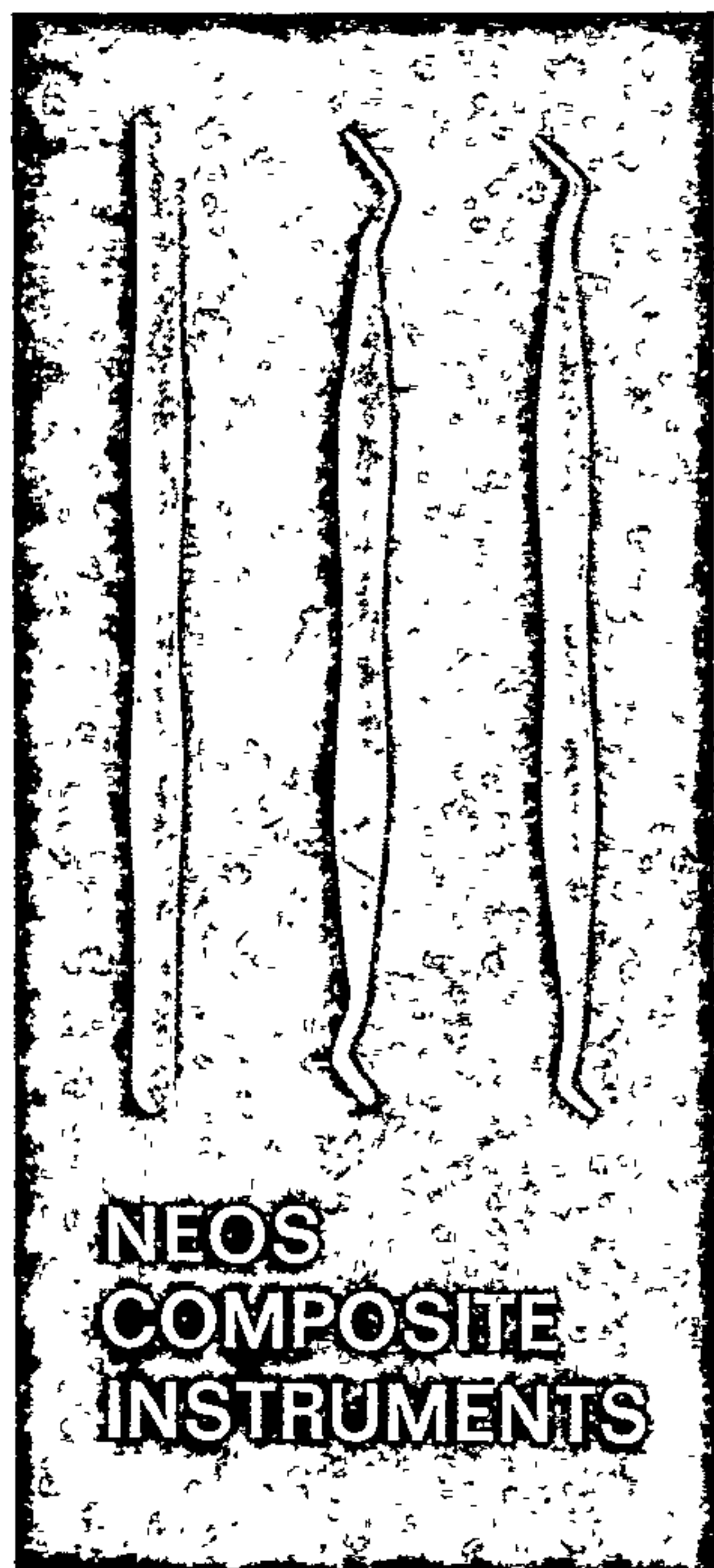
thickness of 0.4 to 1 mm is produced. The palate plate thickness is about one-fifth of this (Fig. 3).

To achieve a sheen, the plate can be plated with gold or rhodium. Additional procedures for finishing are identical to those used for model casting (Fig. 4).

The advantage of electroforming is that it provides exact reproductions of the anatomic conditions. Electrodeposition of metal eliminates the discrepancies which can occur by incorrect or inaccurate casting. An additional advantage is the minimum expenditure of time and material. Schw. ☐

The best of friends fall out, and so — His teeth had done some years ago.

(Thomas Hood)



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電氣鍍金에 의한 總義齒의 金屬口蓋床 製作

—Electroformed Metal Palates for Complete Dentures—

서울대학교 치과대학 보철학교실

김 영 수

총의치 구개상¹⁻³⁾을 제작하는 데에 용액중에서 고 성능의 전류를 이용하여 충격파를 전도시켜 모형상에 녹슬지 않는 강철(stainless steel)을 압힘으로써 얇은 「스테인레스 스틸」판이 모형상에 깔라지게 되는 방법은 또다시 치과의사들의 관심을 불러 일으켜 주었다.

치의학사상 장구한 동안 금속구개상을 제작하기 위하여 금속모형을 만들어 압인하여 만드는 방법(swaging technique)과 주조하는 방법(casting technique)에 대해 열심히 연구되어 왔다.

압인에 의해서 구개상을 제작하는 조작술(두개의 모형과 구개상을 만들기 위한 4~5개의 대응모형을 이용하여 aluminium, silver, platinum, iridio-platinum, palladium-silver, stainless steel 또는 gold alloys 등을 재료로 사용함)은 금속구개상이란 장점에 그 가치를 두고 있다는 점을 강조하고 있다.

1820년에 주석(tin)으로 의치상을 주조한 예가 보고되었고 그 후에 세가지 기본금속(tin, aluminium, gold)을 다른 금속들(silver, copper, bismuth, nickel, magnesium, platinum과 palladium)과 합금하여 시험되었는데 점차적으로 폐기되었으며^{4,6,7)} 요즈음에는 주조구개상은 보통 「코발트-크로미움」(cobalt-chromium) 합금으로 제조한다. 금속구개상을 제작하기 위하여 압인하는 방법과 주조하는 방법의 양자에 소비한 시간과 노력을 생각해 본다면 필요는 없다 하더라도 금속구개상은 의치제작하는데 대한 부수적인 것으로서의 흥미가 있다는 것이 명확하다.

Swenson⁸⁾이 말한 금속구개상의 장점은

Rogers, O.W.: J. Pros. Dent. 23:207~217, 1970.

1. 「레진」(resin)에 비하여 열전도성(thermal conductivity)이 더 좋으며

2. 의치상면의 자극성이 적기 때문에 조직의 내성(tolerance)을 증가시키며 온냉에의 자극을 증대시키고

3. 환자에게 혀가 움직일수 있는 공간(tongue space)을 더 넓혀준다는데 중요한 요소가 되는 구개를 덮는 구개상의 두께를 감소시켜 준다는 것이다.

금속구개상의 단점은 값이 비싸고 「리베이싱」(rebas-ing)과 후구개 폐쇄부를 조절(posterior palatal seal adjustment)하는 경우 유지상의 난점이 있다는 것이다⁹⁾.

「코발트-크로미움」 합금과 같은 비귀금속으로 얇고 커다란 의치상을 주조하는 것은 기술상 복잡한 문제가 관여되어 있기 때문에 비교적 값이 비싸다. 불가능한 문제는 아니나 적합도가 확실성이 부족하며 조절하기 어렵다.

녹슬지 않는 강철(stainless steel)

「스테인레스 스틸」을 전기도금(鍍金)에 의해서 금속의 두께를 형성한다는 것은 현재 사용되고 있는 다른 방법을 이용할때와 마찬가지로 방법은 간단하지만 아직도 각 개인마다의 특유한 방법으로 각구개상을 제작하고 있다는 단점이 있다. 이러한 것은 시간낭비인 것이다. 만약 동시에 많은 수의 구개상을 최소한의 관리로 제작할수 있다면 다량생산하는 방법이 이점이라 할수있다. 이런점을 감안하여 인공재료인 경석교 모형상에서 금속구개상을 전기도금하여 제작하는 방법을 고안해 냈다. 구강내 상태에서 변색되는 것을 방지하기 위하여 금속을 도금할 때에 유도되는 중압이 적은 「니켈」을 도금에 사용하였는데 「니켈」은 금으로 도금하였고 어떤때

에 있어서는 「로듐」(rhodium)으로 도금하였다.

방 법

도금하는 과정에 들어가기 전에 제작모형에 일부변형(modification)을 가해서 복제(duplication)한다. 이와 같은 것은, 첫째로 금속구개상이 의치속에 파묻힐 유지부를 만들어 주고 둘째로는 금속구개상의 양측에 있게 되는 의치상수지에 대한 끝임선(finishing line)을 형성해 주는데 필요하다(그림 1).

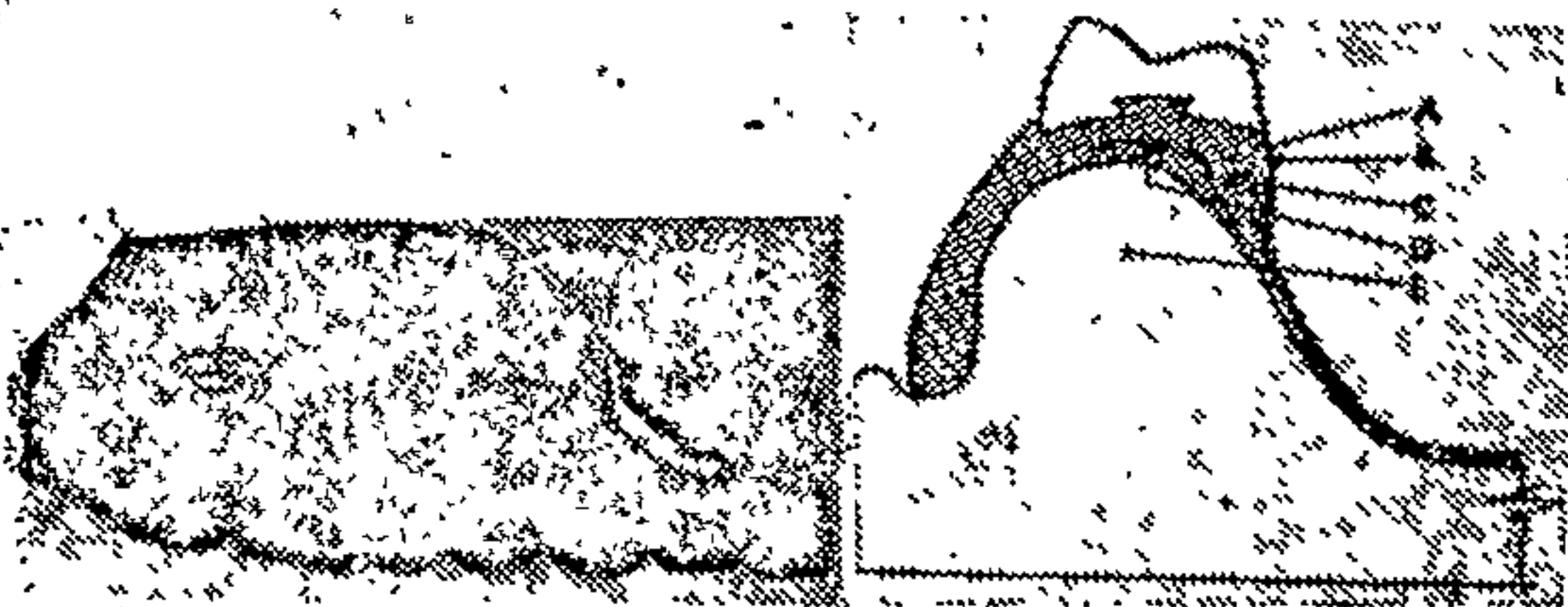


그림 1A. 전기 도금으로 제작된 금속 구개상 의치를 전후관계로 절단한 것임. 「에크리릭 레진」에 파묻힌 금속의 두께가 증가된 것을 주시할 것. 「그 유지부」에 있어서 구개상에 더 강한 힘을 부여하기 위해 이 부위에 금속을 상당히 두껍게 침착시켰음.

그림 1B. 금속 주위에 “acrylic sandwich”가 여하히 형성되어 있는가 하는 것을 보여주는 구치부의 절단면도해. 끝임선은 양쪽 설측부(A)와 조직면(B)에 수지가 두껍게 되도록 와 있다.

- (A) 설측 finishing line
- (B) 금속 도금형
- (C) 구개측 finishing line
- (D) 치조골 정상에 띄워준 부위
- (E) 경석고 모형

1. 반장두께의 「쉬이트 왁스」(sheet wax, dental modeling wax)를 원모형의 치조정상 둘레에 깎는다(그림 2).

2. 잘라낸 「쉬이트 왁스」상에 「코발트 크로미움」의 치상의 끝임선을 주조물에 만들어 주기위해 주조할때 사용하는 기성형태의 재료를 적당하게 배열한다(그림 3).

3. 준비된 원모형을 통법으로 복제하여 모형용 경석고를 부어 넣는다(그림 4).

4. 복제한 모형을 다듬는다.

5. 모형의 기저부에 약 0.5cm길이의 작은 구멍을 2개 뚫는다.

6. 그림 4에 보여주는 바와 같이 전도체부위까지 연결시켜줄 철사를 구부려서 인산아연 「씨멘트」로써 뚫어 놓은 구멍속에 접착시킨다(그림 5).



그림 2A. 반장 두께의 「왁스」를 복제할 모형의 치조정상에 깔음.

그림 2B. 설측의 절단면도해

- (A) 완충납
- (B) 경석고 모형



그림 3A. 기성형태의 재료를 완충납위에 덧붙임. 복제할 준비가 끝난 상태의 모형.

그림 3B. 설측의 절단면도해

- (A) 완충납
- (B) 기성형태의 재료
- (C) 경석고 모형

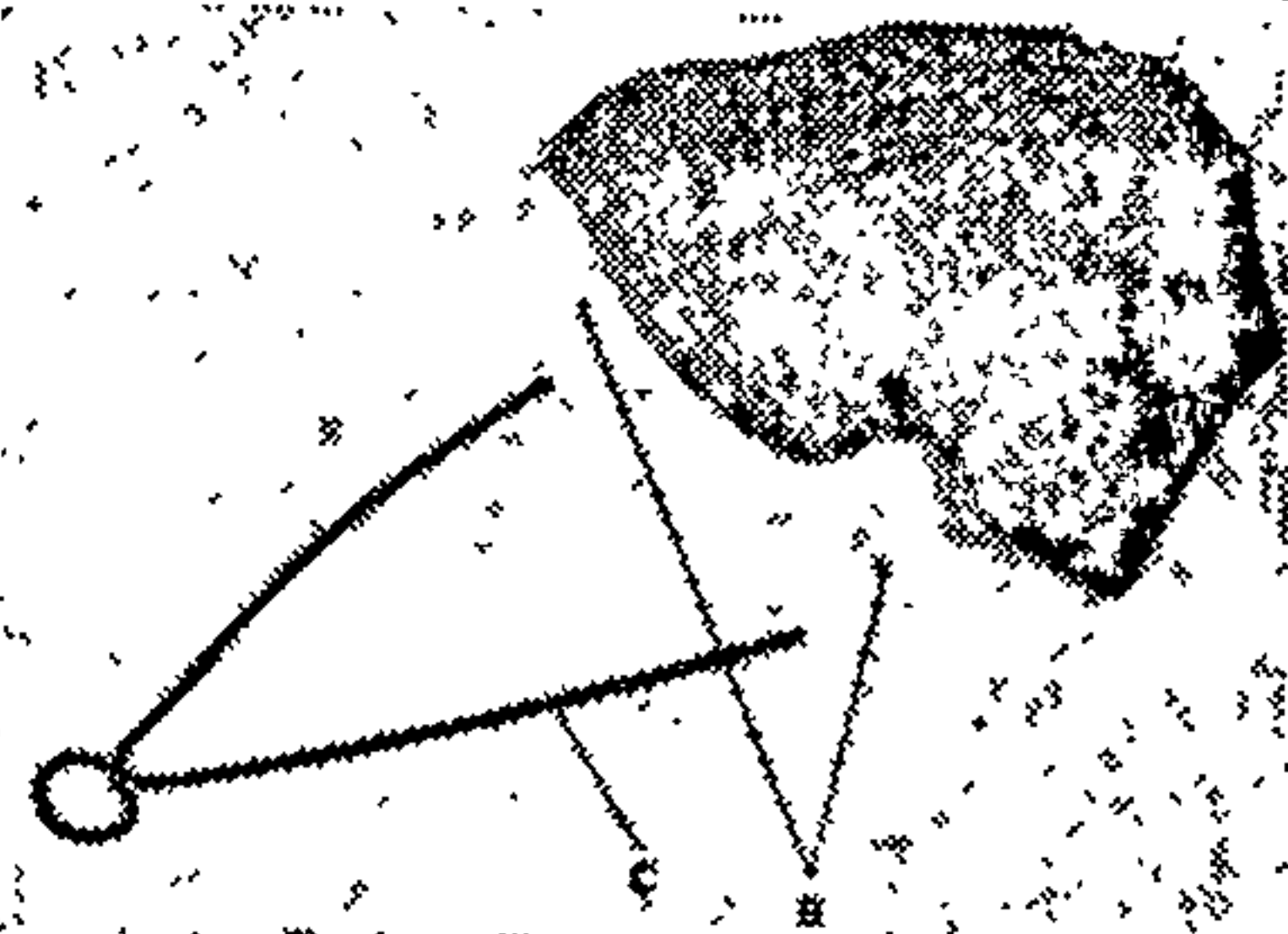


그림 4. 복제된 경석고 모형(A). 전도체부위까지 연결해줄 철사(C)를 삽입하기 위해 모형의 기저부에 뚫은 구멍(B).

복제된 모형의 금속화

전도성있는 은 (Degussa Conductive Silver 200, Degussa-Frankfurt au Main)을 도포하여 모형을 준비한다.

1. 모형의 도금할 부위에 전도성있는 은을 얇게 발라

준다.

2. 전도성 있는 은칠위에 용매제 「이더」(ether)를 다시 칠한다.

3. 모형에 삽입된 철사에도 은「페인트」를 도포하므로써 전류가 도금될 부위로 전도되는 것을 확실하게 한다

4. 경석고 모형중에 처리되지 않을 부위는 노출된 부위에 전해액에 의한 화학적인 영향을 받지 않도록 보호해 주기위해 「에폭시 레진」(epoxy resin)을 도포한다.

전기도금법

1. 온도조절장치가 달린 P.V.C.통(A.C.I. Plastics)속에 8「리터」의 전해용액(Barrett's¹⁰) modified solution, Nickel sulfamate 450g/l, Nickel Chloride 30g/l, Boric Acid 45g/l, PH 3.5—5.0)을 넣는다.

2. 온도를 100°F와 120°F(38°C와 49°C)로 유지시킨다.

3. 음극의 전류밀도를 평방「피트」당 20 내지 60amp.로 유지시킨다. (평방「메시미터」당 2.2내지 6.6amp.를 유지시킨다) 이와같은 방법으로 23개의 구개상을 제작했다(그림 6).

4. 두께가 0.017과 0.045「인치」사이의 구개상의윤과 0.004내지 0.009「인치」되는 구개상을 제작하는 데는 16 내지 18시간 동안 도금한다.

끝 마칩

1. 도금된 모형을 씻어서 건조한다(그림 6).

2. 「세퍼레이팅 디스크」로써 구개의 윤곽을 필요할만큼 잘라낸다(그림 7).

3. 구개상을 경석고 모형에서 들어 올린다(그림 8).

4. 통법으로 연마하는데 보통 금이나 「로디움」도금하기 전에는 연마할 필요없다.

5. 도금한 구개상에 금이나 「로디움」을 도금한다.

6. 경도치(300 D.P.N.)가 증가되었으며 금의 함량(92%)이 높아 졌으므로 전해액에 시안화물(cyanide)함량이 적고 구연산(citric acid)이 가해져서 완성시에 대단히 반짝거리는 광택이 나며 침착된 금이 보통 시안화물 용액을 사용한 것보다 더 단단해지는 금도금용 전해액을 사용해야 한다.

7. 구개상을 원모형에 다시 장착시킨다.

8. 인공치를 배열하고 구개상까지 포함하여 납의치(wax denture)를 형성한다.

9. 납의치를 매몰해서 은성처리하여 정상적인 모양을 형성하여 완성한다(그림 9와 10).

토 론

금속화한다는 것은 모형상에 얇고 연속적인 금속면을



그림 5. 전도체에 까지 연결시켜 주는 철사가 정위치에 「씨멘트」로 접착되었으며 철사로부터 도금될 부분에까지 이르는 전도성면을 형성하기 위하여 금속화 되었음.

그림 6. 중압이 낮은 「닉켈」로써 도금한 후의 모형. 도금된 부의 가장자리에 전류가 집중되기 때문에 한계부에 결절이 생긴다.



그림 7. 전기도금된 구개상을 필요한 형태만큼 「세퍼레이팅 디스크」로써 주위를 돌아가면서 자른다.

그림 8. 전기도금된 구개상을 경석고 모형에서 분리함. 금 또는 「로디움」으로 보호막을 입히고 나면 상악총의치내에 삽입할. 준비가 다 완료된 것임.



그림 9. 전기도금한 금속 구개상의 총의치. 금속과 「에크리릭 레진」에의 끝맺음선을 주시할 것.

그림 10. 전기도금된 금속 구개상이 달린 상악총의치의 기저면. 금속과 「에크리릭 레진」간의 끝맺음 선을 주시할 것. 매몰된 금속의 유지에 보탬이 될 구멍들. 이런 방법은 임의로 하는 것임.

형성하는 방법인데 이렇게 하므로써 전기를 전도시킬수 있다. 거울용 유리에 은을 입히는 데도 사용되는 것과

같은 예의 화학용액으로 부터 은을 침전시키는 은환원법(silver reduction method)과, 그 방법은 비슷하지만 은대신 동(copper)을 사용하는 것으로써 특수한 「프래스틱」(plastics)에 도금하기 위하여 최근에 발전된 전기를 이용하지 않는 동환원법(electroless copper reduction method)이 시험되어 왔다.

이런 과정에서는 모형을 비흡수성으로 만들 필요가 있다. 그것은 「왁스」에 풀이거나 「에폭시 레진」으로 처리하여 만들 수 있는데 전류가 필요치 않은 구리의 경우에는 「메칠 에칠 케톤」(methyl ethyl ketone)에 용해한 A. B. S. (acrylonitrile-butadiene-styrene) polymer로써 처리한다.

이와같은 방법은 도금한 구개상이 변형되어 적합이 불량해 질 수가 있으며 화학약품이 손에 오염될 수 있다고 생각되므로 다른 방법이 고안되었다. 「오일」(Degussa Silver Mixture 112(oil) Degussa-Frankfurt au Main)과 수분(Degussa Silver Mixture C(water base) Degussa-Frankfurt au Main)이 주성분으로 된 약품이 고안되었는데 이런 「페인트」들은 가열 하므로써 금속은 은으로 환원되지만 소요되는 열로 인하여 종종 전도면에 수포를 형성하는 증기가 생겨서 모형이 파괴되는 수가 있다. 그렇지만 도포한 후에 건조기 내에서 건조하고 표면환원을 「컴파운드 토치」(compound torch, torch lamp)의 화염으로 시작하면 좋은 표면을 만들 수 있다.

가장 간단하고 가장 효과적인 방법은 도금을 요하는 면에 전도성있는 「페인트」(Degussa Conductive Silver 200 Degussa-Frankfurt au Main)를 칠해서 덮는 것이다. 이 「페인트」는 모형에 삽입한 철사와 반드시 접촉이 되도록 해서 전류가 도금될 부분까지 전도되어야 한다(그림 5).

전도체의 저항은 절단면(전도체가 얇으면 얇을 수록 그 저항은 커짐)에 의해서 반비례로 변하며 그 길이(전도체가 길으면 길수록 저항은 커짐)에 대해서는 정비례하기 때문에 철사에 접촉되는 면적은 가능한한 크게 해 줘야 한다. 제조업자는 전도성의 전도성을 증가시키기 위해 열처리 할 것을 권장하고 있다. 「페인트」와 함께 용매제 「이더」(ether)를 사용하므로써 확산되는 요소가 증가되며 적합도에 영향을 미칠 수 있는 두께는 감소되며 전도성은 대단히 증가한다. 그러므로 열처리는 필요치 않게 된다. 이용매는 결합제로써 사용되는 「이더 오일」(ether oil)과 유기성분을 제거하며 「페인트」에의 은분자간 접촉을 더 진밀하게 한다.

공업적 생산방법과 같은 「닉켈」 전기도금법은

1. 복잡한 형태라도 제작할 수 있고,
2. 대단히 확실성있게 한정된 좁은 면적과 다양한 표

면의 성질을 재생할 수 있으며,

3. 미세한 점까지 자세하게 형성할 수 있고,

4. 넓은 면을 형성할 때에도 적용할 수 있기 때문에 급속히 증가하고 있다.

이미 사용되고 있는 예로써는 공업용 부분품이 몹새 낡아 마모되거나 오래 쓴 기계면을 개조할때⁴⁾ 전기타자, 「프래스틱」물질을 틀에 넣어 만드는 음각(die)의 제조, 「레이더」와 유도기, 표면조성(roughness) 기준기와 축음기 제조공업중에 「스탬퍼」(stamper)등과 같은 여러가지 주요부분품의 제작등이다.

「닉켈」은 cc당 상대밀도가 8.9gm이란 점이 「코발트 크로미움」과 「스테인레스 스틸」과 비교할 수 있는 점이다. 사용하는 전해액과 조절하는 상태에 따라서 「닉켈」은 $55-110 \times 10^3$ p.s.i.의 최대장력을 갖으며 130-275 D.P.N.의 경도범위와 32-5%의 백분율 신장도를 갖는다. ¹³⁻¹⁵⁾

「닉켈」로 도금침착된 장력과 경도와 연성의 관계는 서로 연관성이 있다. 장력이 증가하면 경도도 증가하는 반면 연성과 백분율 신장도는 감소한다. ¹⁵⁾

덧붙여 말하자면 상기성질들과는 관계가 없는 내부압력이란 것이 있는데 이러한 힘은 변형되거나 전기도금된 금속이 모형에서 분리되는데 충분할 정도로 클 수가 있다. 전기도금으로 침착된 금속은 장력 또는 수축력이 있을 수 있어서 침착된 금속을 굽이 가게 하거나 벗겨지거나 구부러지게 할 수가 있으며 압축 또는 팽창력은 침착물을 부풀어 오르게 하거나 도금을 구부러 뜨릴 수 있다.

watt형의 전해액^{13, 15, 16)}으로 형성된 침착물은 장력이 18,000내지 23,000 p.s.i에 이른다. 이런 압력은 필요는 없지만 도금으로 침착된 금속이 영구적으로 기저 금속에 부착되는데 그다지 해롭지는 않다. 전기도금에는 정확한 재생을 확실시 하기 위해서는 장력이 10,000 p.s.i.를 초과해서는 안된다. 「닉켈」을 도금하는데 사용되는 전해액인 「셀파메이트 배스」(sulfamate bath)의 내부장력은 1,000-6,000 p.s.i 사이이다. ¹³⁾ Kendrick¹⁶⁾은 평방 「피이트」당 50amp.의 전류밀도(5.5amp. per square decimeter)일때 작용하는 전류밀도가 놓여진 「닉켈」 「셀파메이트 배스」(600gm. per liter)는 침착물내에서 14,000 p.s.i의 내부압축력을 나타낸다고 보고했다. 평방 「피이트」(22amp. per square decimeter)당 전류밀도가 200amp. 일때의 침착물은 압력이 없었다.

「셀파메이트 배스」는 사용될 수 있는 조건의 범위가 상당히 넓다. 온도가 25°C에서 최대전류밀도가 평방 「피이트」당 140amp. (15amp. per square decimeter)이며 75°C일때 최대전류밀도는 평방 「피이트」당 (32amp.

per square decimeter) 300amp까지 증가됐다. 이것은 시간당 각각 0.007[인치]가 침착되는 것과 시간당 0.015[인치]가 침착되는 것의 비율이 일치하는 것을 보여주고 있다. 그러나 전류밀도가 높으면 높을수록 음극 면상의 두께의 기복이 더 심해지며 온도가 높을수록 팽창계수는 보상을 요하는 요소로써 나타난다.

Kendrik¹⁶⁾은 평방[피트]당 (22amp. per square decimeter) 전류밀도가 200amp이고 온도가 60°C인 농축된 「셀파메이트」용액(600gm. per liter)으로부터 침착된 압력이 없는 니켈을 만들어 냈는데 그 침착비율은 시간당 0.01[인치]였다.

이 논문에서 기술한 용액에 의한 최대 침착비율은 시간당 0.0025[인치]였는데 조정하는 조건을 개선함으로써 증가시켜줄수 있었다.

어떤 실험에에서는 보통 사용하는 얇고 납작한 형태의 양극(陽極, anode)을 사용하지 않고 도금되는 금속의 두께를 조절하기 위하여 도금될 물체의 형태에 맞춰 모양을 이루어준 양극(contoured anode)을 사용하므로써 일반적으로 구개연보다 후연폐쇄부가 더 두껍게 형성되었으며, 이러한 구개상들은 상당히 견고했다.

최초정에 침착된 「니켈」은 구개면에 있어서 보다4—5배 두꺼웠다(그림 1A). 이것은 혀가 움직일수 있는 공간을 감소시켜 씹이 없이 「아크리릭 레진」에 파묻히는 구개부분에 강도를 증가시켜 준다.

「니켈」과 동(copper)을 교대로 겹쳐 형성하는 일련의 얇은 층으로 된 구개상을 제작하여 보았으나 혼합식 금속 도장법(coating)을 사용한다는 것은 단지 한번쯤 생각해볼만한 흥미있는 공론일뿐이며 기술적인 난점을 더 증가시키는 것 뿐이다.

전기도금은 치과 기공소에서 할수있는 것이며, 적당한 장치만 설립된다면 제작근거로 보아 다수의 구개상을 동시에 제작하는데 이용하는 것이 더욱 적합하다.

결 론

상악총의치의 금속구개상을 전기도금법에 의하여 제작하는 새로운 방법을 기술했다. 전기도금하는 것은 구조하는 방법이나 압인하는 방법보다 면재생에의 정확성이 더 높고, 관여된 과정과 소요의 재료가 적으며 전기도금으로 침착된 금속은 주조시에 일어나는 금속자체의 오차나, 모형과 배움모형에 의한 오차가 일어나지 않는다.

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The electroformed gold matrix inlay technique

O. W. Rogers

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Introduction

Casting by the "lost wax" method has been known and used for thousands of years,⁽¹⁾ but it was not until 1907 that this procedure was given a dental application and the cast gold inlay evolved.⁽²⁾ The advent of the indirect technique, using casts, overcame the difficulties of accessibility and led to the development of crown and bridge and complete mouth rehabilitation procedures. Intensive research has since refined and improved both materials and techniques, but in spite of these refinements and improved technical skills, failures still occur. This is mainly due to the inaccuracies of the impression material, the inlay wax, the die material, and also to inherent errors in the investing and casting procedures. These inaccuracies individually and collectively result in poor marginal adaptation of the completed inlay. As the degree of spinning and burnishing for marginal adjustment is limited, the cast gold inlay would be improved if it had an independent margin with sufficient ductility for accurate adaptation.

The concept of providing an adaptable margin by the use of a gold matrix is not new. In 1887 Charles Alexander⁽³⁾ used a pure gold matrix to construct inlays. Gold foil was burnished directly into the cavity preparation to form a matrix and crystal gold was used to build up the bulk of the inlay. The gold was

removed from the tooth and gold solder flowed between the interstices of the crystal gold and the gold matrix to form the completed inlay. The inlay was then cemented into the tooth.

From the single matrix technique the double matrix technique evolved. One gold matrix was formed to fit into the cavity preparation and the second matrix contoured to form the external surface of the restoration. The space between the two matrices was filled with gold solder.⁽³⁾ Where the two matrices joined at the surface of the cavity an extension of 0.8 mm of pure gold was left. This pure gold periphery was burnished against the tooth when the inlay was inserted.

Gillett and Irving⁽⁴⁾ consider that the slight overlap of the matrix margin is superior to the flush finish of gold foil restorations. The margin can be finished and easily adapted to be left in close apposition to sound enamel areas to provide protection to the cut enamel rods at the cavity surface. Hinman⁽⁵⁾ observed a lower incidence of recurrent caries with this type of gold inlay than with gold foil restorations and Hollenback considers that dental cements show less tendency for dissolution under these circumstances.⁽⁶⁾

The electroformed gold matrix inlay

To combine the advantages of the cast gold inlay and the gold matrix technique a simple efficient method of producing the matrix, even within complex cavity design, was developed. The matrix is approximately 0.01 in. thick,

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(3) Hinman, T. P.—Methods of filling teeth with gold inlays. Items Interest, 28: 1, 1-3 (Jan.) 1906.

(4) Gillett, H. W., and Irving, A. J.—Gold inlays by the indirect system. Brooklyn, N.Y., D. Items Interest Publ. Co., 2nd ed., 1933 (pp. 17-20).

(5) Hollenback, G. M.—Personal communication.

capable of uniting with the cast gold alloys used in inlay construction and eventually becoming an integral part of the completed restoration. The matrix is formed by electro-deposition.

This is a normal indirect inlay technique except that the casting is done into a preformed gold matrix. The internal or tooth-fitting surface is made of pure gold, whilst the remainder of the inlay is cast gold alloy. The periphery of the gold matrix is left extending slightly beyond the cavity outline form. This border holds the matrix firmly in the investment during burning out and casting procedures, and it provides a soft burnishable

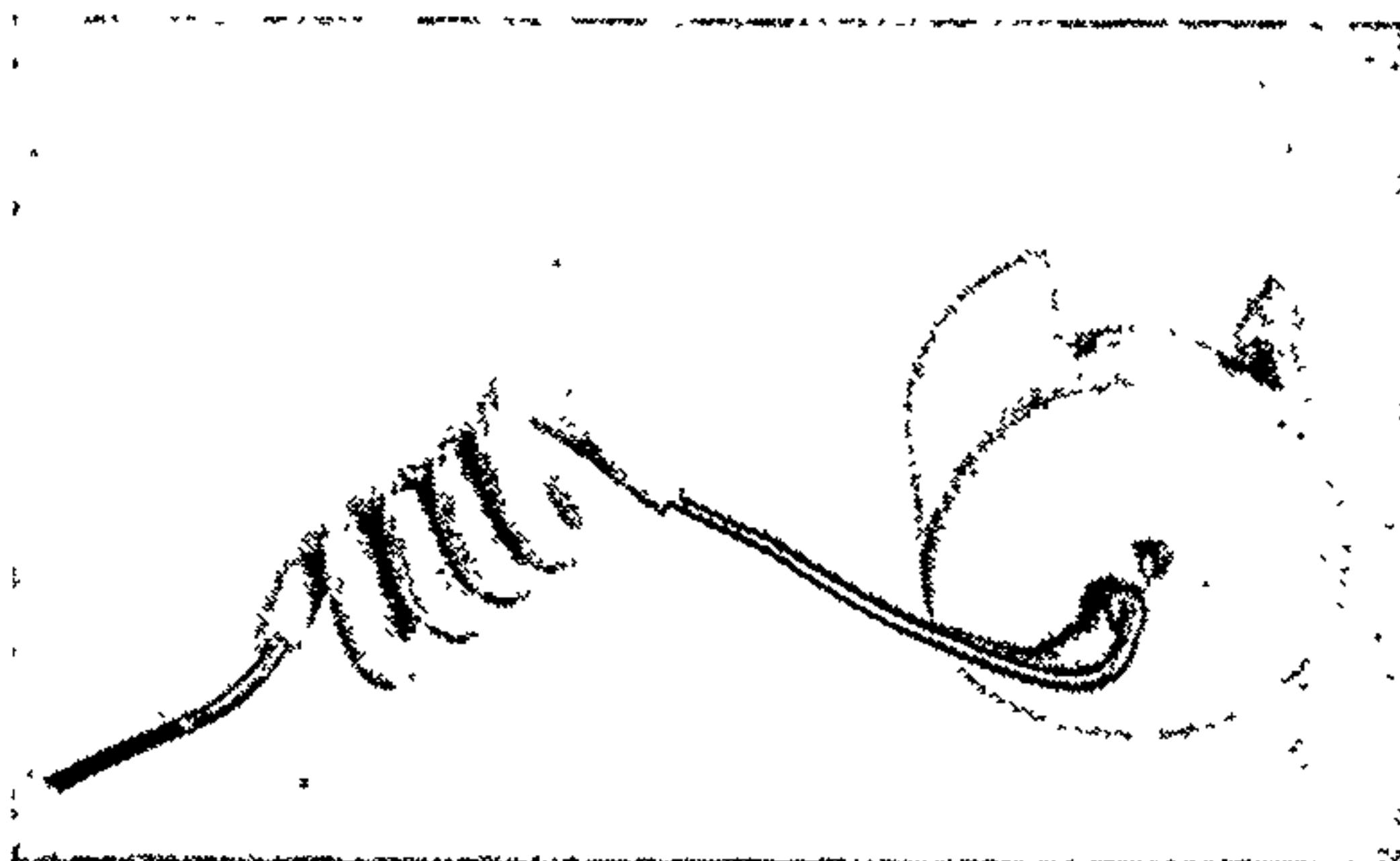


Fig. 2.—The end of the wire is bent for additional retention prior to cementation.

impression is used again to construct the dies for electroforming the gold matrices. Only the teeth being restored are reproduced in this second step and their supporting structures are excluded. Hydrocal is preferred for these dies since it is difficult to remove the electroformed gold matrix from a Densite die.

The detailed cast for construction of the gold inlays and individual dies of the tooth for the electroforming procedure are produced from the original impression (Fig. 1). The base of the individual Hydrocal die is smoothed with a garnet disc and all sharp edges beyond the cavity outline are rounded. A hole is cut in the base of the die $\frac{1}{8}$ in. deep, by using a round tipped fissure bur in the air-rotor. Four inches of insulated wire, stripped at both ends to expose $\frac{1}{2}$ in. wire with one end bent at right angles (Fig. 2), is cemented into the base of the die with zinc phosphate cement (Fig. 3a) in preparation for metallizing.

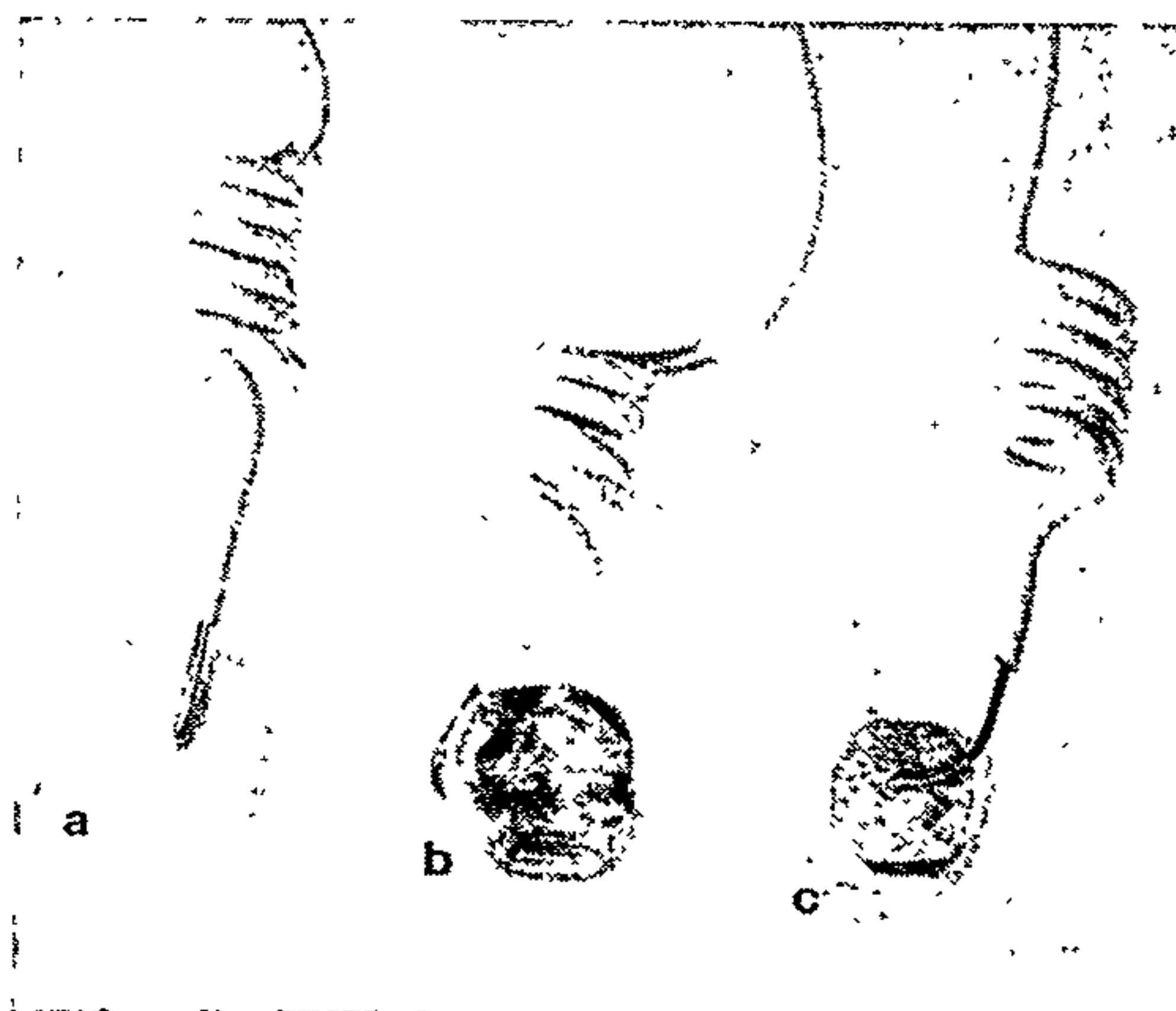


Fig. 3.—(a) Wire cemented in place. (b) Matrix outline established in metallizing paint. (c) Metallizing paint continued around the base of the die and over the inserted wire.

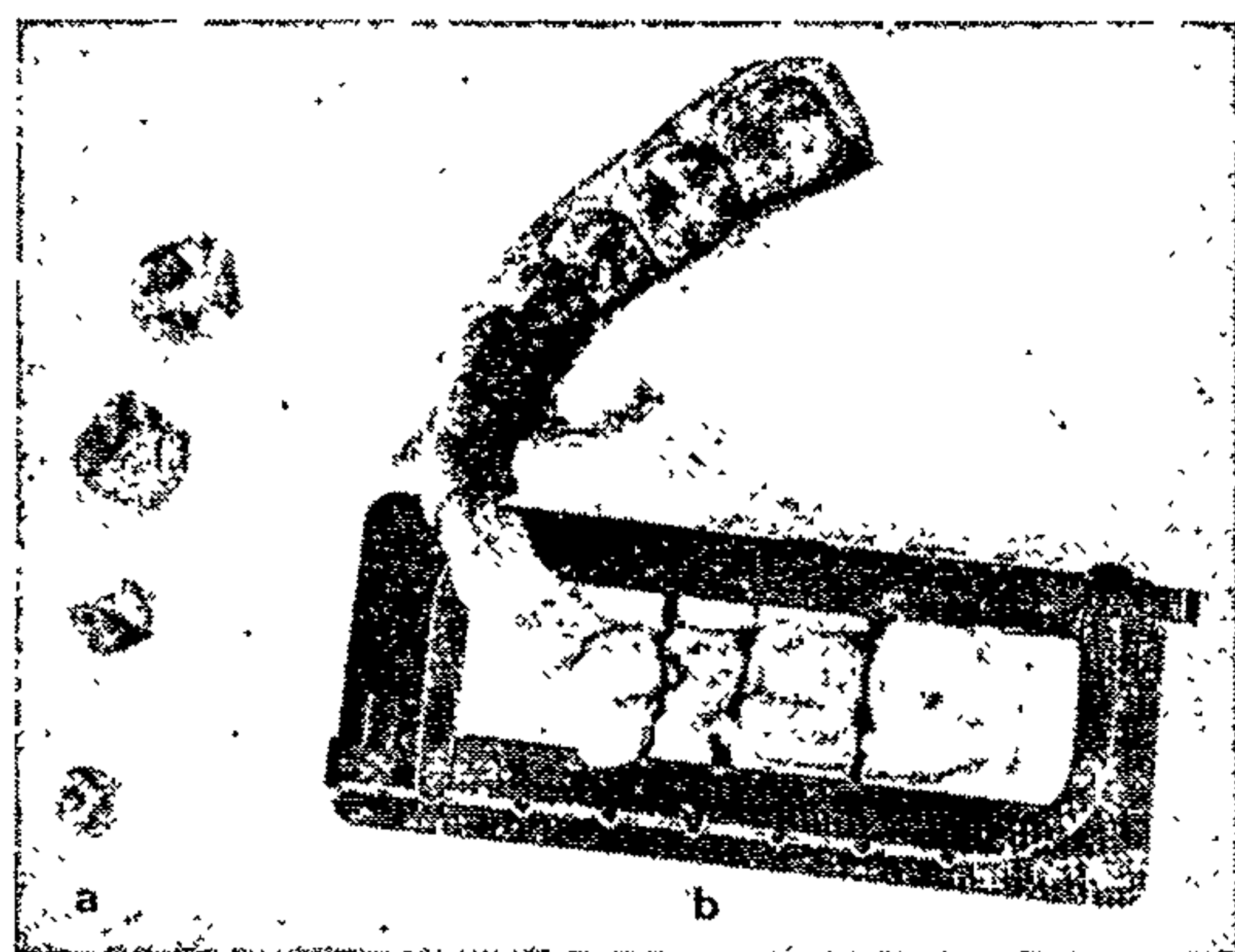


Fig. 1.—(a) Individual Hydrocal dies used for electrodeposition of the gold matrix. (b) Di-Lok tray and Densite master cast used for indirect inlay procedure.

margin around the completed inlay ensuring marginal adaptation. Investigations were carried out to develop:

- (1) a method for metallizing the surface of artificial stone to make it electrically conductive;
- (2) an electrolyte that would provide heavy deposition of gold, have a high rate of deposition, and require little maintenance, and
- (3) the casting conditions necessary to produce bonding between the cast gold alloy and the electroformed gold matrix.

Impression procedure

If polysulphide rubber or silicone impressions are used, an individual tray is recommended.⁽⁶⁾ After pouring the Densite working cast, the

⁽⁶⁾ Rogers, O. W.—Electrodeposited copper individual impression trays. J. Pros. Den., 16: 1, 19-25 (Jan.-Feb.) 1966.

Metallizing

The usual materials and methods used for making impressions conductive for electroforming copper or silver dies⁽⁷⁾⁽⁸⁾⁽⁹⁾⁽¹⁰⁾⁽¹¹⁾ are not satisfactory for electroplating gold on artificial stone. The simplest and most effective method found was with conductive silver paint.* The viscosity of this material tends to reduce the spreading factor and the metallizing paint builds in the line and point angles of the die. This limitation is improved by the use of solvent ether which ensures a closer contact between the silver particles in the paint; since the spreading factor is increased and the surface distribution improved, the thickness of the paint is reduced. It was also found that the heat-curing treatment as recommended by the manufacturer is unnecessary.

The conductive silver paint is applied to the Hydrocal die with a fine camel-hair brush. The initial application is made onto a flat surface, such as a pulpal or axial wall, keeping the paint away from the line angles. If this is not done the paint tends to concentrate in these areas and to round off the sharp cavity detail. The brush is dipped in ether and applied to the paint on the die. The silver paint spreads readily and by the continued application of ether, a minimal thickness of silver maintains sharp detail of the cavity (Fig. 3b).

After the desired matrix shape is established, the surplus silverizing paint is worked onto the base of the die. The base area, including the bare section of the wire, is coated and broad contact established with the matrix outline form (Fig. 3c). Because the resistance of a conductor varies inversely with the cross-sectional area and is directly proportional to its length, the width of the thin silver conductive surface must be exploited to ensure the conduction between the wire and the matrix

area; for example, with the M.O.D. preparation, the proximal surfaces are extended down and along the base to the wire inserted in the die. The metallized area has a circular form around the die.

To prevent wasteful deposition of gold, the metallized surfaces beyond the actual matrix outline are covered with a non-conductive paint. Thus, only the matrix area presented a conductive surface for electrodeposition. Nail varnish is a satisfactory non-conductive paint and its definitive colour provides accurate demarcation of the boundaries.

Electroplating

The electrolyte was that used by Rogers and Armstrong⁽¹²⁾ and had the following formula:

Potassium gold cyanide	14.1 g
Potassium cyanide	18.3 g
Potassium carbonate	14.1 g
Boric acid	11.4 g
Distilled water	to 1 litre.

The electrolyte at an operating temperature of 65°C and a current density of 30 amp per square foot (3.2 amp per square decimeter) has a rate of deposition of 0.004 to 0.005 in. per hour. Agitation is necessary and a gold anode is used.

The area of the metallized surface exposed must be determined to calculate the current required for electroforming. Since there is a number of surfaces involved it is simpler to mark out each area on graph paper so that the total area can be assessed visually. A M.O.D. cavity on a bicuspid has an approximate metallized area of $\frac{1}{2}$ square inch, and the recommended current density is 208 milliamps per square inch. Therefore, a current of 69 milliamps is required for the M.O.D. die. Because of polarization the anode must always be four times the area of the cathode, otherwise the electrolyte is depleted of gold ions.

It is possible to electroform more than one die at a time, and with the equipment and method described⁽¹²⁾ nine or ten dies can be electroformed concurrently. It is vital that electrodeposition should commence immediately the dies are placed in the electrolyte, otherwise the free cyanide in the electrolyte will

⁽⁷⁾ Skinner, E. W., and Phillips, R. W.—The science of dental materials. Philadelphia, W. B. Saunders Co., 6th ed., 1967 (p. 424).

⁽⁸⁾ Cannon, R. W. S.—The electroforming of dental models with silver. Austral. D. J., 9 : 2, 77-81 (Apr.) 1964.

⁽⁹⁾ Phillips, R. W., and Dettman, F. J.—A study of some variables associated with copperplating of dental impressions. J. Pros. Den., 6 : 1, 101-113 (Jan.) 1956.

⁽¹⁰⁾ Tuckfield, W. J.—Principles and techniques of electrodeposition as applied to dental practice. Austral. J. Den., 42 : 1, 6-27 (Jan.) 1938.

⁽¹¹⁾ Rosenstiel, E.—Improvements in electroforming dental dies. Brit. D. J., 88 : 10, 269-275 (May 19) 1950.

* Degussa conductive silver 200.

⁽¹²⁾ Rogers, O. W., and Armstrong, B. W.—Electroforming a gold matrix for indirect inlays. J. Pros. Den., 11 : 5, 959-966 (Sept.-Oct.) 1961.

attack the thin layer of silver metallizing paint, destroying its continuity and also contaminating the electrolyte.

A plastic jig is used to mount the dies and gold anode so that they can be connected to the rectifier before they are placed into the plating bath. The jig is constructed from $\frac{1}{2} \times \frac{1}{4}$ inch strips of perspex and is rectangular in shape. The length of the rectangle is made to sit on the top of the plating tank and the width of the rectangle is three inches—the anode-cathode ratio (Fig. 4).

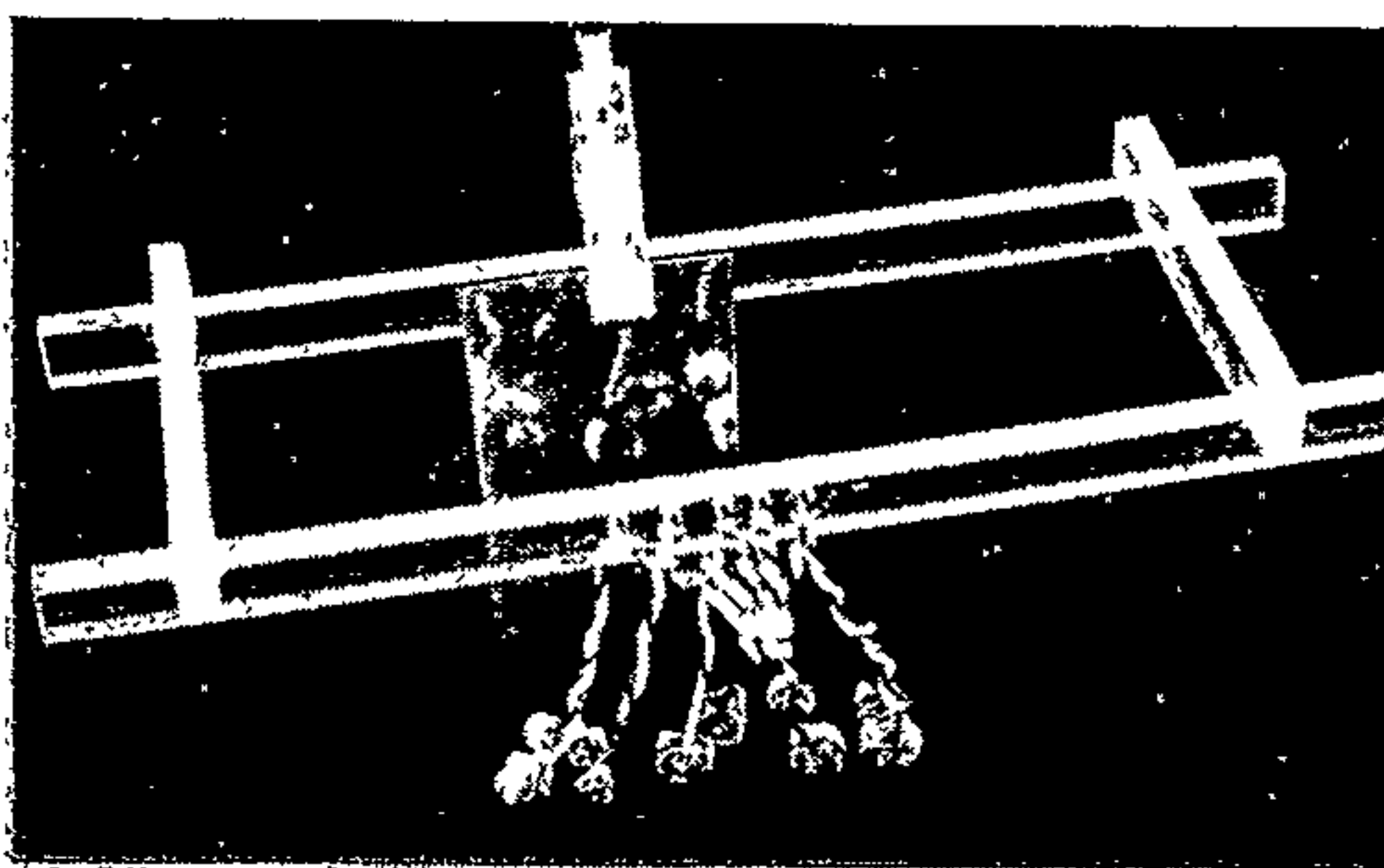


Fig. 4.—The plastic jig used to establish the correct anode-cathode distance. The dies (cathode) and the gold (anode) are arranged for depth and distance apart before placing in the electrolyte for electroforming.

The gold anode is attached to one of the long arms of the jig at its predetermined depth and is held in place with an "alligator" clip. The dies are positioned on the other long arm of the jig with the metallized surface facing the anode. The dies are held in position by wires placed through holes drilled in the plastic, and the wires are twisted on each side of the plastic jig to hold the dies firmly in position.

When the electrolyte in the plating tank is 65°C, the agitating mechanism is started. The anode and cathode leads are connected and the rectifier is turned to a low setting. The jig is placed into its position on the tank and the rectifier adjusted to the calculated amperage. Electrodeposition occurs immediately and the silver metallizing paint is not attacked by the cyanide content of the electrolyte.

The colour of the electrodeposited gold should be bright yellow. A brown deposit indicates too high a current density, insufficient agitation, too low temperature, or a depletion of free cyanide ions in the electrolyte. The dies are removed from the electrolyte after two and a half hours, then thoroughly washed and dried and inspected.

Removal of the matrix from the die

The matrix outline is cut out with a separating disc, leaving a narrow margin approximately 0.5 mm extending beyond the outline form of the cavity. This over-extension of the gold matrix forms the periphery used for marginal adaptation. The gold matrix is removed from the die by heating in a Bunsen flame until it reaches dull red heat, and is then plunged into water. The Hydrocal disintegrates and releases the matrix. Sometimes when a large surface area is involved, the steam generated by heating raises the pulpal floor of the matrix. This is corrected later during the adaptation procedure. The carefully washed gold matrix is boiled in 50 per cent hydrochloric acid to destroy any vestiges of cyanide and after rinsing in water the matrix is ready for adaptation.

Adaptation of the gold matrix

The gold matrix, now in an annealed state, is placed on the original Densite working cast and burnished into intimate contact with the die. Particular attention is paid to the adaptation of the gold margin.

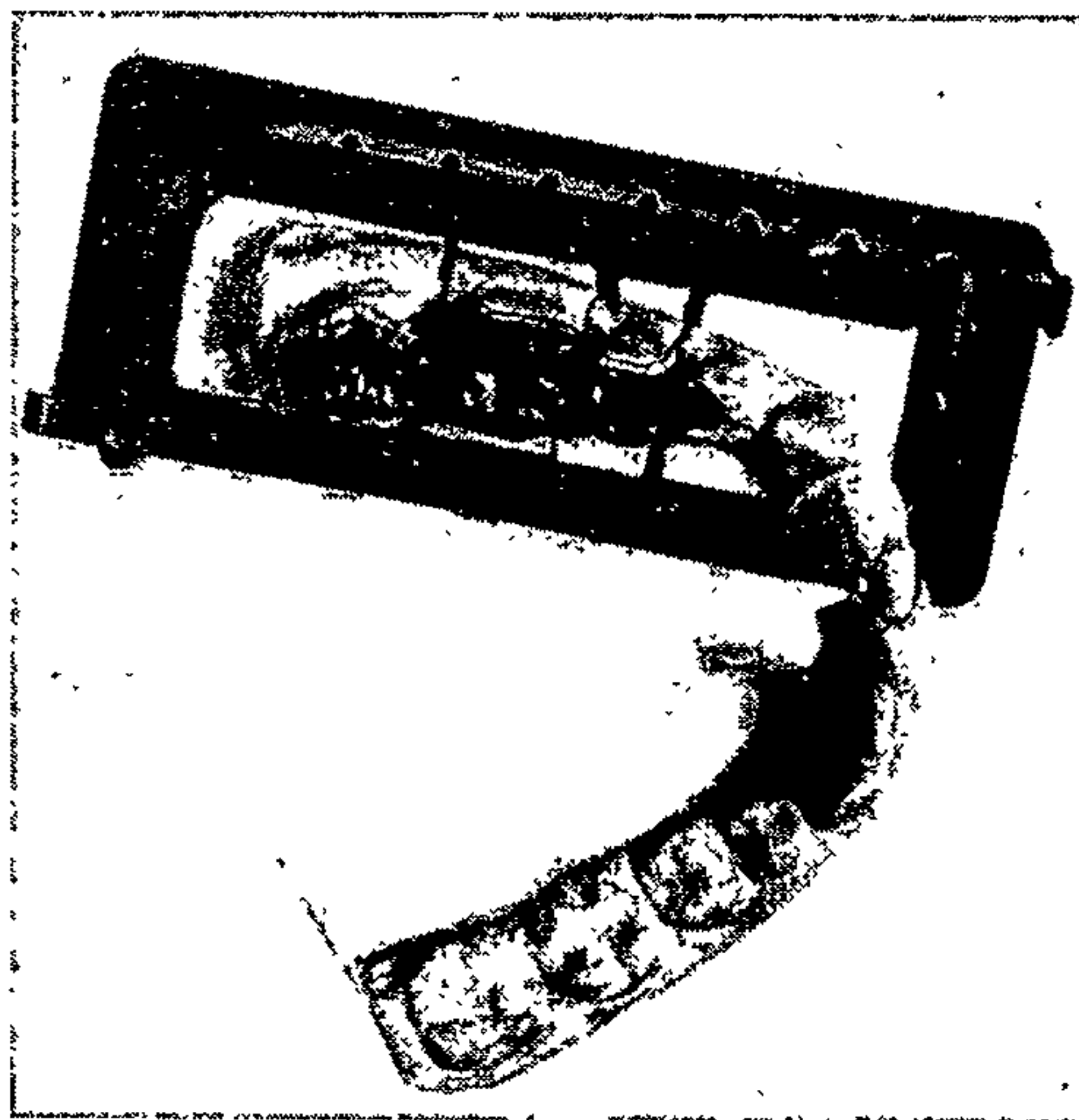


Fig. 5.—Inlays waxed up within the electroformed gold matrix. Note the gold border extending beyond the wax.

The wax pattern

The inlay wax is contoured and carved within the gold matrix and thus is reinforced against the intrinsic or extrinsic forces producing distortion (Fig. 5). Manipulative procedures not possible with conventional wax

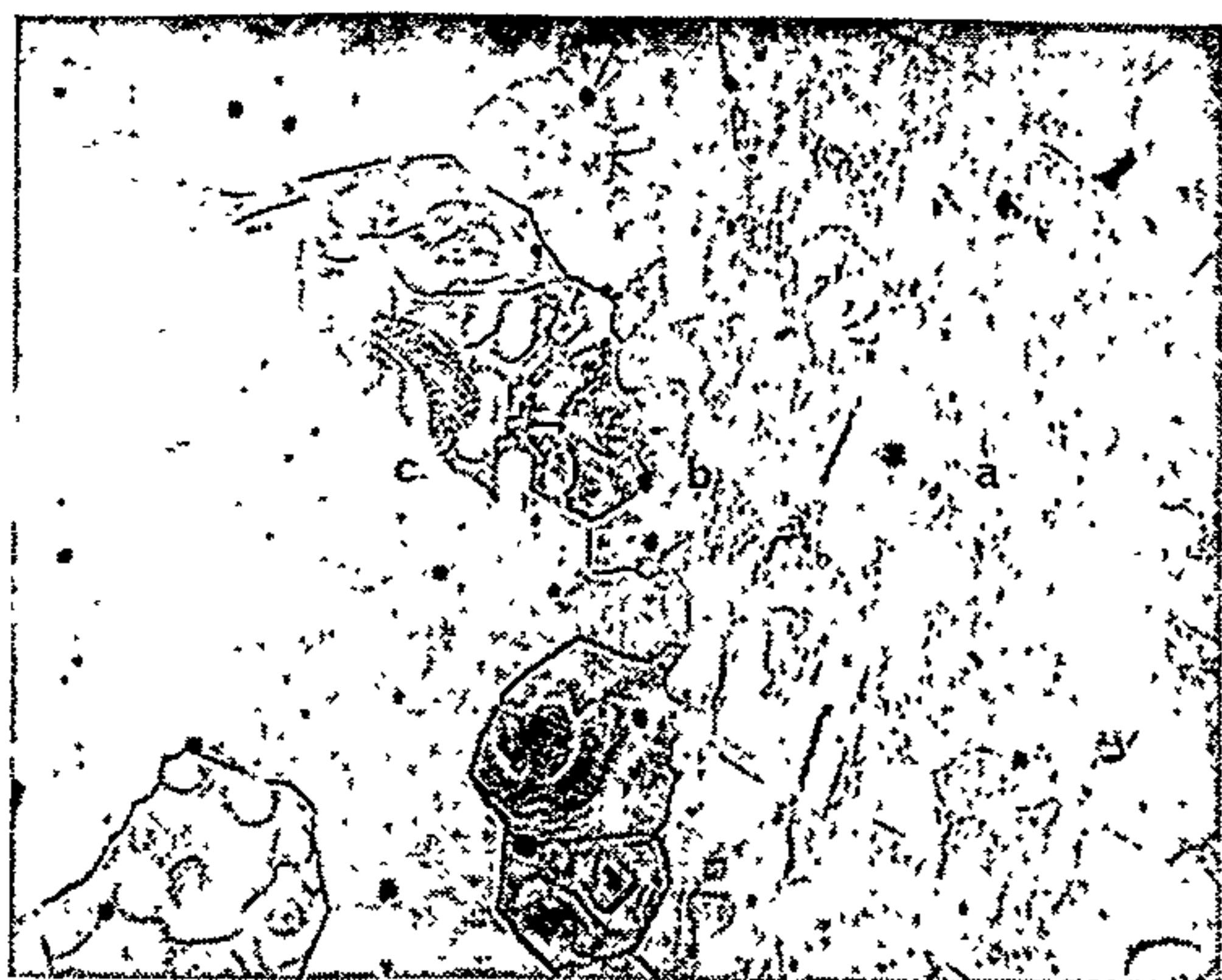


Fig. 6.—Type C gold alloy cast against electroformed gold matrix (a); interface (b) between alloy (c) and electroformed gold. $\times 800$.

patterns may be carried out with these patterns. As the wax is completely reinforced and the margin of the pattern is formed by the gold matrix, the matrix inlay pattern can be returned to the tooth preparation to check and adjust the contact points and the occlusal surface under functional conditions. The gold margin can be burnished into intimate contact with the tooth.

Investing, wax elimination, and casting

Variations in casting procedures, mould and casting temperatures with the use of different gold alloys were studied and 44 specimens were prepared, electrolytically etched, and photomicrographs were taken. The results of these observations will be published separately,

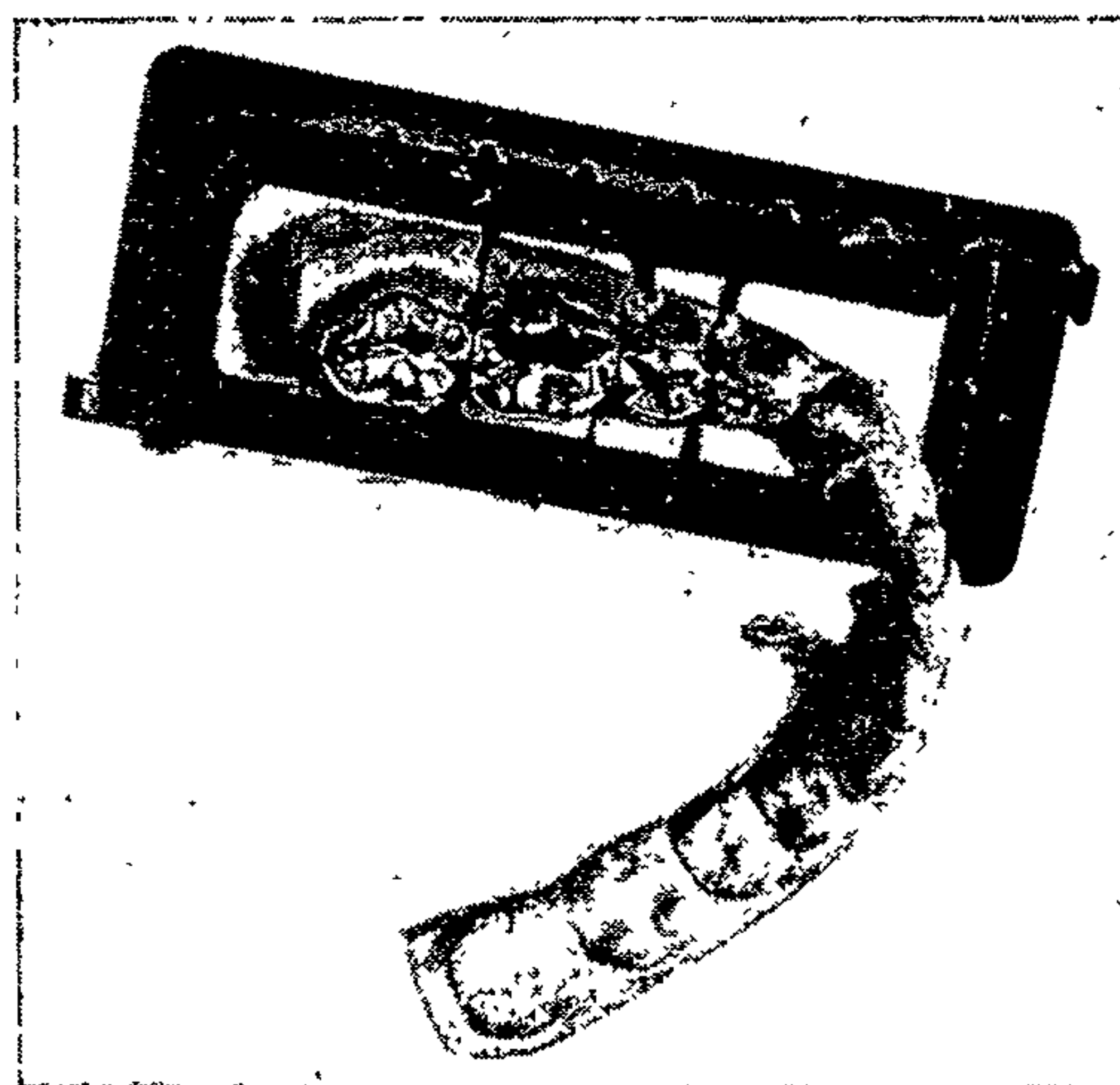


Fig. 7.—The completed gold inlays returned to the master cast for marginal adaptation.

but for the purpose of this paper it can be accepted that excellent union between the electroformed gold and the cast gold alloy occurred with evidence of diffusion, providing:

- (1) a mould temperature of 700°C is used,
- (2) a casting temperature of 22.5°C to 50°C above the liquidus of the gold alloy is established, and
- (3) a heat saturation time of 5 minutes is provided before quenching.

Figure 6 shows the type of bond that exists between the cast and electroformed golds. The interface (b) between the cast gold alloy (c) and the electroformed gold (a) shows a strong bond with wetting and diffusion, and inclusions are minimal and only of a gaseous nature. This form of pin-hole porosity is common to all dental castings.⁽¹³⁾

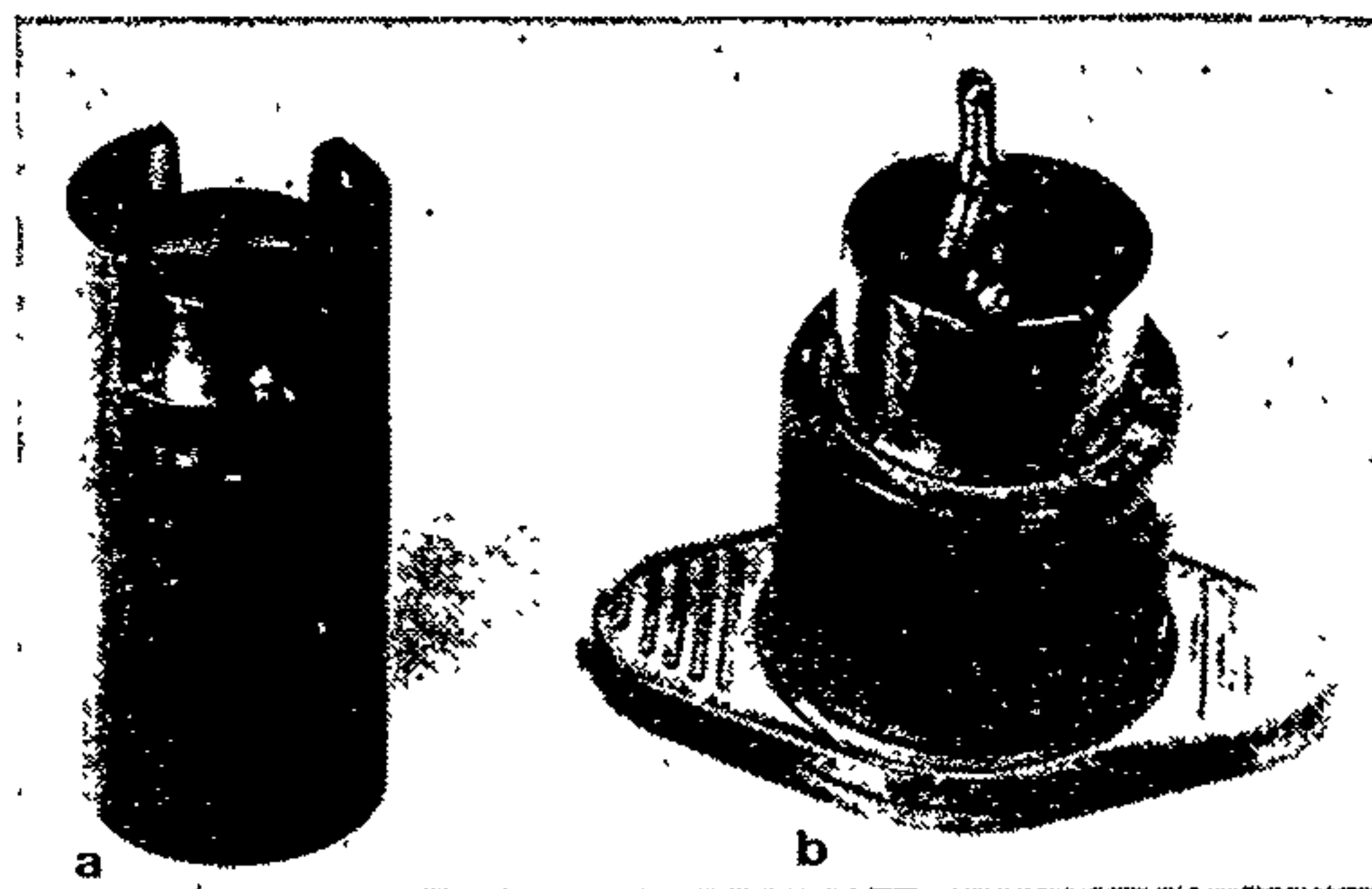


Fig. 8.—(a) Standard M.O.D. die. (b) Distorted die with a casting in place.

The wax pattern is sprued, invested and a casting made using the thermal expansion technique. The inlay is cast at a mould temperature of 700°C with a Type B or C gold alloy. The casting is removed from the investment, pickled in acid and polished, care being taken not to damage the soft gold borders (Fig. 7).

Pinlays

The gold matrix technique can be used for pinlay construction. After the die is metallized, precast hard gold alloy pins are inserted into the die. The gold alloy pins must contact the metallizing paint but the exposed heads of the pins must be free from metallizing paint. During the electroplating, gold deposited on the exposed heads unites the pins with the gold

⁽¹³⁾ Skinner, E. W., and Phillips, R. W.—The science of dental materials. *Op. cit.* (p. 388).

matrix. Due to the leverage possible on the junction of the pin and the gold matrix it is considered advisable to strengthen this area by gold soldering before taking the matrix from the die. The pinlay is waxed to contour and cast with a hard gold alloy.

Cementation of the restoration

As the internal surface of the inlay is formed by the gold matrix, casting irregularities are limited to the external surface of the inlay. After establishing correct occlusal relationships, contact points, and marginal adaptation, the gold alloy is heat treated and the inlay cemented into place. The soft gold margin is finally burnished before the cement is set.

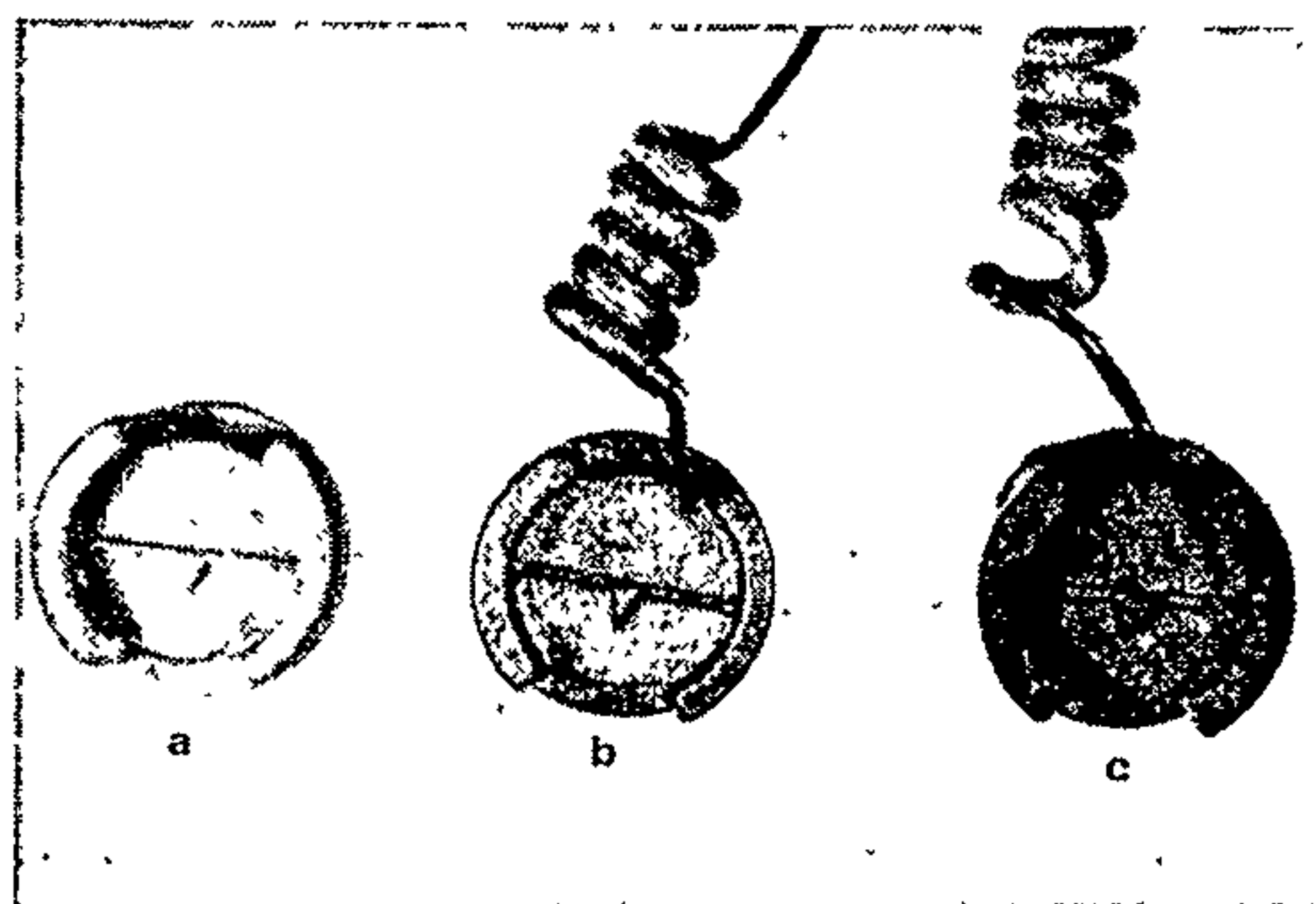


Fig. 10.—(a) Hydrocal cast of distorted die. (b) Cast metallized. (c) Cast electroformed with gold.

Discussion

Casting into an electroformed gold matrix combines the desirable properties of hard gold alloys with ductile, and therefore adaptable margins. The possible use of even Type D alloys could lead to reassessment of resistance form and consequently greater tooth conservation.

The microstructure and the properties of strength of dental castings are essentially established when the alloy achieves complete solidification. Ady⁽¹⁴⁾ considers it advantageous to obtain the best qualities possible during the solidification of the alloy, as subsequent heat treatment to strengthen the casting is done at the expense of the ductility required to burnish the margins.

The use of smooth rotary instruments instead of abrasive or cutting instruments has been

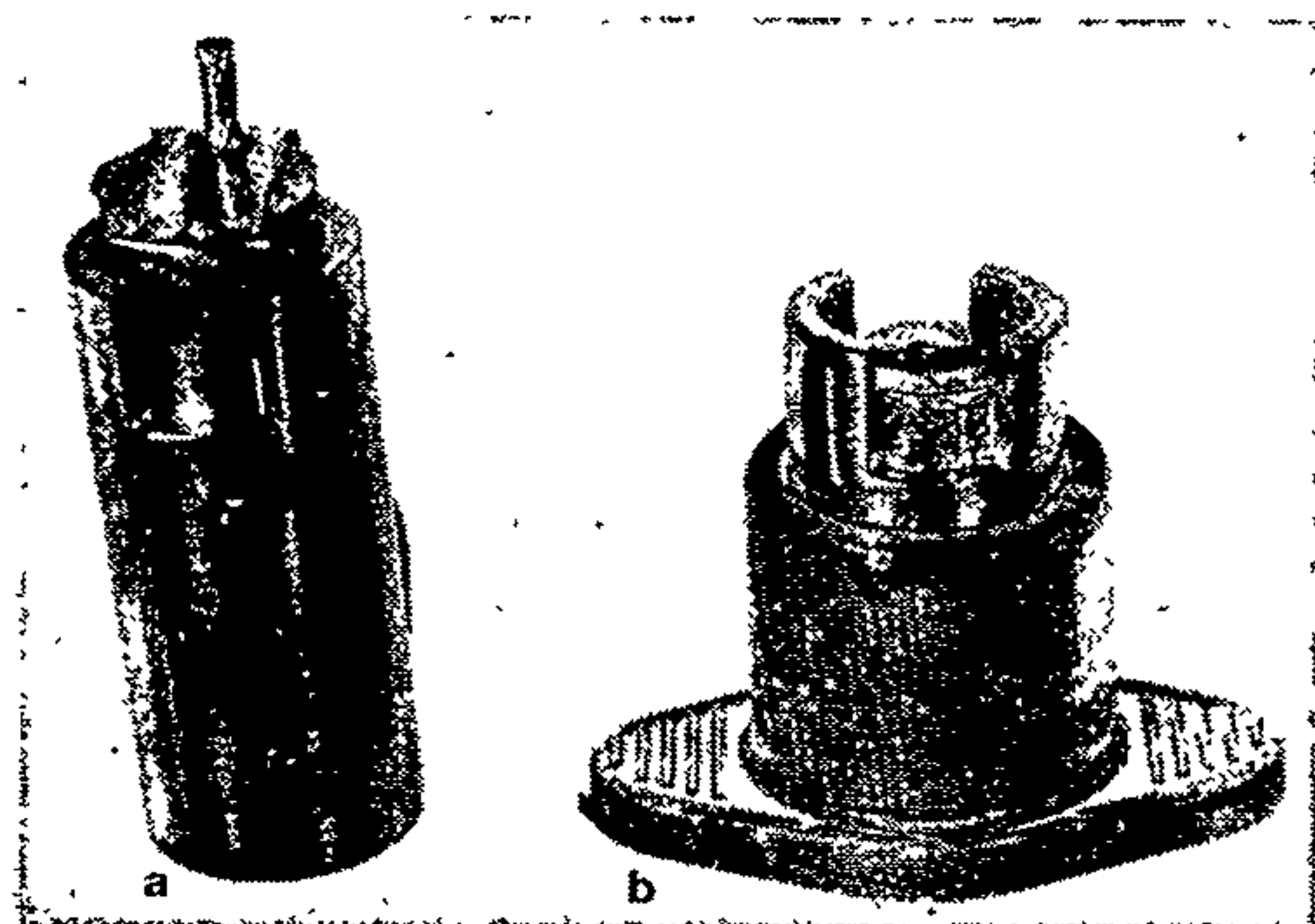


Fig. 9.—(a) Casting placed on the standard die showing the amount of distortion existing between the dies. (b) Distorted die.

With most inlays it is found that burnishing the gold margin prior to cementation gives such good retention that removal is difficult. The ability to adapt the soft margin into undercut areas, such as the infrabulge area, should be exploited when considering the outline form of the cavity.

Figure 8 shows a standard die and a casting seated in a distorted version of the standard die. A crossline is placed on the pulpal floor of the distorted die for identification (Fig. 9). The extent of the distortion is seen when the casting is placed on the standard die.

An impression is taken of the distorted die and a Hydrocal cast made (Fig. 10). The electroformed gold matrix when removed from the cast accurately fits the distorted die (Fig. 11b). After annealing and burnishing the gold matrix is adapted to fit the standard die (Fig. 11a). The gold matrix is waxed to contour and the inlay cast. The completed inlay fits the standard die (Fig. 12).

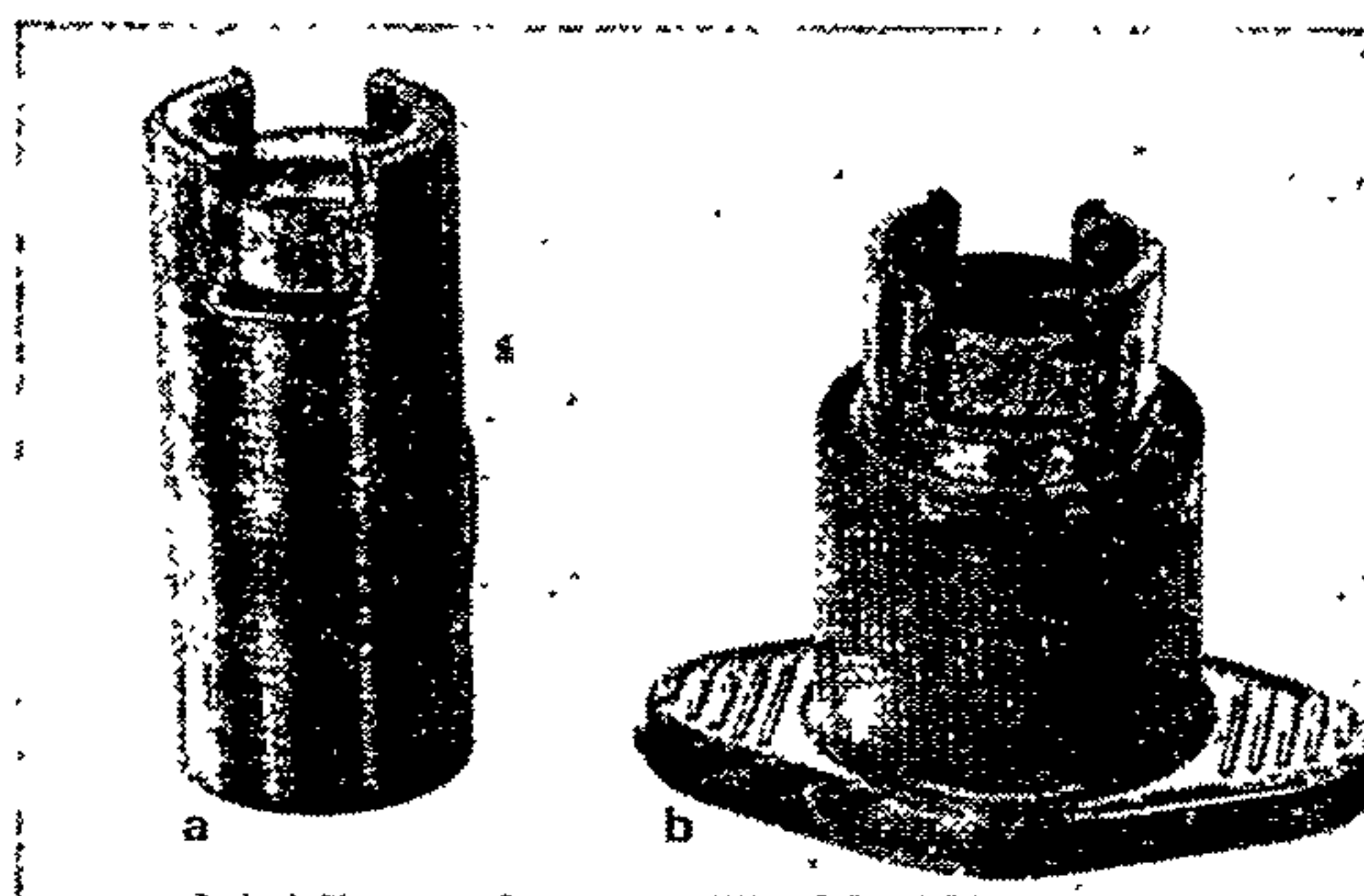


Fig. 11.—(a) Electroformed matrix burnished to the standard die. (b) Electroformed matrix prior to burnishing showing its original fit to the distorted die.

⁽¹⁴⁾ Ady, A. B.—Effect of solidification time on the microstructure and physical properties of dental gold casting alloys. J. D. Res., 45 : 3, 921-926 (May-June) 1966.

advocated to burnish Type B and C gold alloys.⁽¹⁵⁾ A well formed strong margin with the grain deformation, slip lines and surface deformation resulting in work-hardening was obtained with deficiencies up to 70 microns. This technique is limited by access and such work-hardened margins would be brittle.



Fig. 12.—Completed casting made from the distorted die placed in the standard die. This is possibly due to the adaptability of the electroformed gold matrix.

Another technique for marginal adaptation⁽¹⁶⁾ is to over-extend the wax pattern and then after casting to selectively grind and burnish the inlay margin to a silverplated die. A gold with Brinell hardness of less than 84 must be used and the bulk of the inlay increased in preference to increasing the hardness factor of the gold.

Margins have been closed past the reported film thickness of zinc phosphate cement,⁽¹⁷⁾ and studies have reported film thicknesses of 3 to 9 microns, which is well within the limits of 40 microns acceptable for cementation.⁽¹⁸⁾

⁽¹⁵⁾ Eames, W. B., and Little, R. M.—Movement of gold at cavosurface margins with finishing instruments. *J.A.D.A.*, 75: 1, 147-152 (July) 1967.

⁽¹⁶⁾ Krug, R. S., and Markley, M. R.—Cast restorations with gold-foil-like margins. *J. Pros. Den.*, 22: 1, 54-67 (July) 1969.

⁽¹⁷⁾ Christensen, G. J.—Marginal fit of gold inlay castings. *J. Pros. Den.*, 16: 2, 297-305 (Mar.-Apr.) 1966.

⁽¹⁸⁾ Norman, R. D., Swartz, M. L., and Phillips, R. W.—Studies on film thickness, solubility and marginal leakage of dental cements. *J. D. Res.*, 42: 4, 950-958 (July-Aug.) 1963.

These points emphasize the importance of marginal adaptation to inlay procedure and the desirability of having a soft gold margin independent of the cast gold alloy. The cast gold alloy can be heat-treated when and if required, and the margin of the electroformed gold matrix inlay is responsive to burnishing into intimate tooth contact with a minimum thickness of cement.

The additional retention possible by burnishing the matrix into infrabulge areas overcomes one of the problems associated with inlay restorations. The retention of the inlay is no longer entirely dependent on the cementing medium.

In the technique described the matrix is electroformed on a second die made from the original impression and then returned to the master cast for completion of the inlay. Minor discrepancies usual to the indirect procedure are compensated for by burnishing and spinning the margin of the matrix prior to cementation of the inlay.

Compensation for hidden undercuts or indefinite margins that are apparent when the working cast is examined is possible as the annealed matrix can be placed in the original tooth preparation and burnished into intimate contact with the tooth. An undercut area within the cavity preparation will expand the annealed matrix as it is seated. If the undercut area is not involved during burnishing procedures the matrix should withdraw. Alternatively, the undercut area can be cemented out, or the cavity preparation redesigned before burnishing. To assist the removal of the matrix, sticky wax can be luted onto its external surface before taking the impression. The impression is taken with the burnished matrix in position, the matrix removed with the impression, and a new working cast poured. The inlay procedure is then continued.

As previously mentioned, the problem of wax distortion is eliminated as the inlay wax is confined within the gold matrix. The strength of the matrix makes handling possible and, if necessary, the occlusal surface and contact points can be checked functionally and adjusted. The margins of the pattern will not be damaged as they are part of the gold matrix.

The narrow gold margin that extends about 0.5 mm beyond the cavity outline must be placed beyond direct occlusal forces. Just as marginal adaptation is possible, the margin

could be displaced by direct occlusal loading. For this reason the cusps are capped so that the cast gold will protect the soft gold margin. The success obtained with the single and double matrix inlays of the past confirms that the margins will endure if thought is given to their positioning. Three-quarter veneer restorations on anterior teeth do, however, provide a problem as it is difficult to minimize the amount of gold visible and to protect the matrix.

The behaviour of the gold matrix in the investment during the casting procedure can only be assumed. Empirically, there is no problem since any discrepancy would be of a sufficiently low order and compensated for by marginal adaptation.

The hygroscopic expansion technique was not used as the mould temperature of 427-428°C was inadequate for diffusion of the cast gold into the electroformed gold matrix.

The electrolyte is a cyanide solution and must be handled with care. Many dentists use a silver cyanide solution for electroplating impressions, so the additional use of a gold cyanide solution is quite feasible. Cyanide

electrolytes must never come in contact with acid solutions, and particular attention should be paid to washing the hands whenever contact is made with the plating bath. Disposable paper towels are preferred for wiping the hands and for the elimination of any spilt electrolyte.

Summary

A modification of indirect inlay technique, using a pure gold matrix prepared by electro-deposition on die stone, is presented.

The matrix is used either in the die or in the cavity for the waxing stage of the pattern.

When cast, the pure gold matrix becomes part of the inlay; this can be made of hard type gold and provides a soft gold margin which can be burnished to the cavity margin. The technique can be applied to pinlay preparations.

The soft gold margin must be protected from occlusal stress. Care must be observed when handling the cyanide electrolyte. Further developments leading to cyanide-free electrolytes are awaited.

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