

**AN EVALUATION OF THE COLOURS OF BASE METAL ALLOY OXIDE
LAYERS, AND ITS EFFECTS ON THE SHADE OF BASE METAL ALLOY
METAL-CERAMIC RESTORATIONS**

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28/11/1997

APPROVED FOR FINAL SUBMISSION

----- DATE 14 November 1997
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DISSERTATION SUBMITTED IN COMPLIANCE WITH THE REQUIREMENTS FOR
THE NATIONAL MASTER'S DEGREE IN TECHNOLOGY: DENTAL TECHNOLOGY,
IN THE DEPARTMENT OF DENTAL TECHNOLOGY, TECHNIKON NATAL

31 JANUARY 1997

I, Grant Brandon Somers, declare that this dissertation represents my own work, both in
conception and execution.

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DEDICATION

This study is dedicated to:

The Somers family, who have allowed me to accomplish all my dreams and who have supported me with love, understanding and guidance throughout my life.

ACKNOWLEDGEMENTS

The author wishes to acknowledge, with sincere thanks, the following people, who through their dedication and patience, have made this dissertation possible:

- Mr Neil Waddell, my supervisor, for his instruction, guidance and motivation to complete this dissertation, and for his commitment to the study and his work in general.
- Dr Dirk Coertze, my co-supervisor, for his methodical input in the study.
- The Higher Qualifications Committee, Technikon Natal, for their financial assistance.
- The Centre for Enterprise Development, Technikon Natal, for their support and assistance.
- The Foundation for Research and Development, for their financial assistance to purchase the materials used in this study.
- The Department of Dental Technology of Technikon Natal, for the use of their equipment and laboratories to carry out this study.
- Romatex Textiles and their laboratory staff, for their patience and friendly service during the use of their Spectrophotometer.
- Somers Engineering, for the manufacturing of the depth gauge.
- Dawn Greef for formatting and printing the dissertation.
- Linda Coertze for proof reading the document.

ABSTRACT

Globally, difficulties are being experienced by the dental world to match manufactured prosthetic restorations with natural tooth colour.

The primary objective of this study was therefore to evaluate the colour of five different metal-ceramic base metal alloy oxide layers, and the effects of each oxide layer on the shade of the porcelain applied to its surface.

The increase in the cost of gold alloys in the dental industry has influenced manufacturers to provide alloys which are cost effective and have adequate mechanical properties for the manufacture of metal ceramic restorations.

Since their initial development, there has been many formulations of base metals alloys. One consistent flaw to these base metal alloys has been the inclusion of base metal elements which turn the oxide foundation dark after firing. This phenomenon is often noticed in the areas where the ceramic is very thin, for example around the cervical areas of a restoration. This darkening of the cervical area directly affects the colour of the gingival margin. The result is that the free gingiva takes on a dark appearance which is not aesthetically acceptable.

Five base metal alloys used in the fabrication of metal-ceramic restorations were selected. The selection of the base metal alloys was based on popularity in the industry, availability and cost.

The following five base metal alloys were selected for the investigation:

- Wiron 99 (nickel-chrome)
- Ceradium V (nickel-chrome-beryllium)
- Remanium CD (nickel-chrome)
- Wirobond (cobalt-chrome)
- Heranium (nickel-chrome)
- Bond-on-4 (low noble content alloy {control group; which was gilded})

Thirty specimens for each alloy group (180 in all) were cast, using the lost wax technique. Three ceramic layers (notably the opaque, dentine and a glaze layer) were applied to the 20mm in diameter surface of each specimen.

A certain school of thought that recommends that the metal surfaces of semi-precious metals be gilded with a gold gilding paste before the application of ceramics in order to block out the dark oxide colour and enhance the colour of the ceramic to a more aesthetic and warmer colour. Therefore, prior to the opaque firing of the control group (Bond-on-4), a gilding layer of Spectra Seal Paste was applied to the surface of each specimen. Following each application of ceramic, the appropriate firing cycles were used to fire the ceramic.

Finally both sample groups were tested against a custom shade guide the colour being compared specifically with the Vita A1 shade. The reason for the inclusion of the shade guide was that, clinically, most shade selections are made by comparing the patient's tooth shade with a manufactured shade guide. A wide selection of shades are available to the ceramist.

The following factors affected the selection of the shade of ceramic used in this study:

- the shade which is most influenced by the dark oxide layer
- the more popular shades of ceramic (the A class of shades).

It was established, through a review of the literature (Crispen, 1991), that the most objective form of testing colour at the time of the study, was the use of a reflectance spectrophotometer. This form of testing had been proved to be cost effective and was consequently used in this investigation.

The study found that the base metal alloys produced darker restorations than the shade guide and gilded control group, and that the differences in the final colour were significant. In terms of hue and chroma, the gilded control group was closer to the shade guide than the base metal alloys. Of greater importance was the fact that there were no significant differences among the hue and chroma of the five selected base metal alloys.

It was found that the oxide colour of base metal-ceramics did not affect the final colour of the restoration, and that the opaque layer effectively masked out the influence of the dark oxide colour. However, it was concluded that, if the hue (the basic family of colour) is significantly different when compared to the hue of a competitive metal ceramic alloy, then the resultant colour of the final restoration will be significantly different.

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CHAPTER ONE

THE PROBLEM AND ITS SETTING

1.1 INTRODUCTION

Since the inception of base metal alloys into the local industry, ceramists have complained that their restorations have been affected by the intermediate oxide layer found on most of the base metal alloy surfaces after firing, and that the shade of their final restoration does not match the shade guide. The shades that are affected the most seem to be the lighter shades, notably the A1, B1 and C1 shades, as per the Vita Shade Guide System (Vita Zahnfabrik, Germany). The effect of these oxide layers is more apparent in the cervical regions where the application of thick body porcelains is not possible due to limited space.

The literature states (McLean, 1980; Phillips, 1982; Yamamoto, 1985) that oxides of base metal alloys are continuously produced during the firing cycles of the porcelain. When the mechanisms of the chemical bond were studied, it was apparent that the oxides that were formed on the alloy surface dissolved into the porcelain. Consequently it was assumed that the effects of the increase in the production of the base metal oxides upon further firings, notably the dentine and glaze firings, would be to influence the shade as a result of the oxide dissolving into the porcelain and therefore coming through to influence the colour of the restoration. Assuming therefore that this assumption was correct, and based on the knowledge that there is a large variety of base metal alloys available on the South African market, it was argued that there was a need to establish which of the most popular brands of base metal alloy would have the least influence on the lighter shades of ceramic. The result would be of great use to the local ceramist. However, the evaluation of colour or shade by the human eye is highly subjective, and it was therefore necessary to devise a more objective method to evaluate the colour of ceramic restorations. It was consequently decided to use a reflectance spectrophotometer in order to establish the differences in the shades of the sample groups. This investigation was possible because a reflectance spectrophotometer was available in the local industry and the expertise existed to use the equipment and to interpret the results.

1.2 THE PROBLEM AND ITS SETTING

1.2.1 THE STATEMENT OF THE PROBLEM

The purpose of this investigation was to evaluate the colours of the base metal alloy oxide layers of selected metal-ceramic base metal alloys available in South Africa in terms of oxide colour after firing, and the effects of these oxide colours on the shade of base metal alloy metal-ceramic restorations in order to establish how base metal alloy oxides relate to the shade guide used during the fabrication of metal-ceramics restorations.

1.2.2 SUBPROBLEMS

1.2.2.1 SUBPROBLEM ONE

The first subproblem was to identify the colour of the base metal alloy oxide layers after firing in order to relate the oxide colour to metal-ceramic restorations.

1.2.2.2 SUBPROBLEM TWO

The second subproblem was to assess the role of base metal alloy oxides with reference to the individual porcelain layering in ceramic production in order to illustrate how base metal alloy oxide colours impact on the colour of the final restoration.

1.2.2.3 SUBPROBLEM THREE

The third subproblem was to validate base metal alloy restorations with reference to the shade guide colour classification in order to establish how base metal alloy oxides relate to the shade guide used in the fabrication of metal-ceramic restorations.

1.2.3 HYPOTHESES

1.2.3.1 HYPOTHESIS ONE

It was hypothesised that the relationship between base metal alloy oxides and the shade guide existed and that the differences in colour could be demonstrated.

1.2.3.2 HYPOTHESIS TWO

It was hypothesised that the type of relationship between the shade of porcelain after each individual layering of the ceramic and the base metal alloy groups, would affect the final colour of the ceramic restoration.

1.2.3.3 HYPOTHESIS THREE

It was hypothesised that metal-ceramic base metal alloys would produce excessive oxide of undesirable colour which would dissolve into the porcelain during the firing process with the result that colour matching to the shade guide would be impeded.

1.2.4 THE ASSUMPTIONS

1. It was assumed that the casting procedure would not influence the resultant colour of the oxide layers.
2. All firing of ceramics would be conducted on a Vita Vacumat 300 (Vita Zahnfabrik, Germany) which incorporates the following features to facilitate accurate firing cycles:
 - calibration of the instrument
 - computerised software which allows for accurate monitoring of the firing cycle
 - programmable memory which recalls original settings
 - highly accurate temperature thermostat.

Consequently it was assumed that the porcelain firing cycles to be used to process the specimens for the different stages of enamelling would be exactly the same.

3. A custom built depth gauge, especially manufactured for this study was used to gauge the depth of the applied porcelain. It was assumed that the criteria for the manufacturing of the depth gauge, notably:
 - accurate digital readings
 - fine adjustment to determine the exact depth of ceramic
 - highly precision engineering

would produce repeatable specimens in terms of the depth of ceramic.

4. A Texflash Datacolour V.2 (Datacolour Colour Future, U.S.A) reflectance spectrophotometer was used to test the specimens during this study and based on the guaranteed accuracy of this instrument, the following assumption was made:
 - although the specific spectrophotometer used in this study (Texflash Datacolour V.2) is used in the textile industry, it was assumed that it would be suitable for the colourmetric testing of dental ceramic.
5. It was evident in the literature that the oxide formation of base metal alloys is continuous during the different ceramic firing cycles (McLean, 1980; Phillips, 1982; Yamamoto, 1985). Consequently it was assumed that the effects of the increase in the production of the base

metal oxides upon further firings, notably the dentine and glaze firings, would be to influence the shade as a result of the oxide dissolving into the porcelain, and that this assumption was also applicable to other brands of porcelain. The following assumptions were made with reference to the formation of the base metal oxide layer:

- the oxide layer would not be adequately masked out by the opaque ceramic layer
 - the control group which would be gilded with a gold gilding material would adequately block out any undesirable oxide colour
 - the gilded semi-precious control group (Bond-on-4) would be suitable as a control, i.e. simulating the role of the shade guide.
6. It was assumed that the thickness of the base metal specimens (0.4mm) would produce the same amount of oxide as produced by a metal-ceramic coping.

1.2.5 DELIMITATIONS

1. This study measured only the effect of oxide colour on the shade of the porcelain, and did not investigate the effects of:
 - different porcelain firing cycles
 - different firing temperatures
 - varying thickness of porcelain layers
 - different methods of condensing the porcelain.
2. Due the time and cost constraints of the study, the investigation was limited to five metal-ceramic base metal alloys available in South Africa. A detailed description on the selection of the base metal alloys is presented in section 3.4.2.1. The following base metal alloys were selected:
 - Wiron 99
 - Ceradium V
 - Heranium
 - Remanium CD
 - Wirobond
 - Bond-on-4 (semi-precious metal{control group})

The selection of these base metal alloys was based on the following factors (Table 3.1):

- popularity
 - availability
 - cost
 - composition.
3. There are many different types of porcelain available on the South African market. One of the most commonly used porcelains in South Africa is Duceram (Ducera, Gesellschaft mbH, W. Germany). The main reasons for Duceram's popularity, are its cost effectiveness and availability. Consequently only Duceram porcelain of the shade Vita Lumin Vacuum A1 was used on all the specimens.
4. The Vita Lumin Shade guide has a rounded tooth shape that is not suitable for accurate colour measuring in the spectrophotometer. Therefore a custom all ceramic shade guide of the same shape and dimensions as the specimens was constructed from the same brand of porcelain as that used for the specimens. All the results were compared with this all ceramic shade guide. A similar method was used by Muia (1982:32) when recommending the production of a custom shade guide.

1.2.6 DEFINITION OF TERMS AND CLARIFICATION OF CONCEPTS

1.2.6.1 METAL-CERAMIC

Metal-ceramic refers to a dental restoration in which the porcelain is fused directly to a cast alloy crown shell that fits the prepared tooth. Also called a porcelain-fused-to-metal restoration (Jablonski 1982:678).

1.2.6.2 METAL-CERAMIC BASE METAL ALLOY

A metal-ceramic base metal alloy is an alloy developed for porcelain bonding and which falls within the following classification:

- Nickel-Chromium: Composed of up to 80% nickel and a chromium content in the 13 to 22% range. In some instances containing up to 2% beryllium by weight (Phillips 1991:365).
- Cobalt-Chromium: Composed of up to 68% cobalt and a chromium content in the 25 - 27% range. They do not contain beryllium (Phillips 1991:366).

1.2.6.3 SPECTROPHOTOMETER

A spectrophotometer can be classified into the following two groups:

- liquid solution spectrophotometers
- solid state reflectance spectrophotometers.

A clear distinction was made between these two types of spectrophotometers in this study. The spectrophotometer used in this investigation was a solid state reflectance spectrophotometer (Texflash Data Colour V.2, Data Colour future, U.S.A) and has been defined by O' Brien (1988:301) as follows:

“A spectrophotometer which scans the entire visible wavelength range (or spectrum) of light reflected from a sample. It provides the information from which it is possible to determine reflectance curves as a function of wavelength for each sample.”

1.2.6.4 OXIDE

Oxide refers to any compound of oxygen with an element or radical (Jablonski 1982:574).

For the purpose of this study it refers to the oxide formed on the surface of the base metal alloy which diffuses into the glass phase of the porcelain to bring the porcelain into atomic contact with the metal surface.

1.2.6.5 CONCEPTS USED TO DESCRIBE COLOUR

Any colour in the visible spectrum is defined by the following three terms:

- **HUE** : the basic family of colour (determined by wavelength) e.g. blue, green, yellow, red.
- **VALUE** : the relative lightness or darkness of a colour. The quality of grey. (NOTE: Y% [capital Y] should not be confused with the [y] co-ordinates used in the chromaticity diagrams.)
- **CHROMA** : the intensity or concentration of the hue.
- **Y%-VALUE** should not be confused with value in the literal sense, for example:

Y%-VALUE : the relative lightness or darkness of a colour (the quality of grey).

1.2.6.6 TRISTIMULUS VALUES

X, Y and Z: Amounts of the three referenced colour stimuli, in a given trichromatic system, required to match the colour of the stimulus considered (ISO/CIE 10526).

1.2.6.7 CHROMATICITY CO-ORDINATES

x, y and z are ratios of each set of three tristimulus values to their sum (ISO/CIE 10526).

1.2.6.8 POSSIBLE CAUSES FOR THE CHANGE OF COLOUR IN DENTAL PORCELAIN

The effects of firing of porcelain are experienced in industry as having two consequences:

- (a) GREENING is defined as: Overfired porcelain will lose grain boundary and become greenish in colour (McLean, 1980:56).
- (b) DEVITRIFICATION is defined as: The effects of repeated firing of high expansion porcelain, causing the porcelain to appear white (Phillips, 1982:515).

1.2.6.9 CHROMATICITY DIAGRAM

A chromaticity diagram is a plane diagram in which points specified by chromaticity co-ordinates represent the chromaticities of colour stimuli (ISO/CIE 10526).

CHAPTER TWO

REVIEW OF RELATED LITERATURE

2.1 INTRODUCTION

The ensuing review of related literature is aimed at providing the reader with insight into the global difficulties that the dental world experiences with matching manufactured prosthetic restorations to natural tooth colour, specifically in terms of metal-ceramic restorations. The review will then continue to describe the increase in popularity of base metal alloys in the commercial environment and the possible reasons for the increased use of these alloys in preference to semi-precious and precious alloys.

An overview of the problems associated with the shade matching of natural teeth to the shade guide and in turn to the metal-ceramic restoration is provided. The reviewed literature will deal specifically with the non-precious metal alloys which produce darker oxides than the previously used precious alloys. The problem of dark oxide surfaces on non-precious substructures has been addressed with the introduction of gilding materials which duplicate the surface lustre that precious metals boast. This will be reviewed in this section as well as in the economics section to follow.

The metallurgy of base metal alloys is reviewed, specifically that of nickel-chrome and cobalt-chrome alloys. In addition, that of beryllium or non-beryllium containing alloys are discussed, in lieu of their oxide formation characteristics.

The last section examines the instrumentation used in previous studies, and offers logical argument for the use of specific instrumentation (i.e. objective evaluation of colour) as opposed to visual analysis using the human eye. In conjunction with instrumentation, there are many different types of colour charts and formulae which previous researchers have used, and these are reviewed in terms of their advantages and disadvantages in relation to this study.

To conclude, a summary of the research done up to the time of this study, and an explanation as to why further research needs to be done on base metal oxides and the effects that these oxides have on the final shade of metal-ceramic restorations, are given.

2.2 NATURAL TOOTH COLOUR VERSUS THE COLOUR OF CERAMIC RESTORATIONS

2.2.1 CERAMIC HISTORY

The first signs of porcelain restorations appeared in the 1800s and with the advent of gold-alloy casting techniques the race was on to find a suitable restorative metal which could be used in dental appliances (Williams 1990:41). In the mid-1960s however, porcelain was fused to metal to enhance the aesthetic abilities of the dental restoration and this led to the production of many different types of base metal alloys used today.

With the rising cost of precious metals and the demand for stronger alloys that withstand all the production processes within the laboratory, the interest in base metal alloys is increasing. These alloys have been introduced onto the market in ever increasing quantities and with claims for their reliability often unsupported by any scientific evidence (McLean, 1980:32). The lack of reliability has stimulated research on how to improve base metal alloy restorations.

2.2.2 A GLOBAL ISSUE

All over the world dental technicians and dentists alike have realized that there is a need to find harmony between the natural tooth and the manufactured prosthesis. Yamamoto (1985:221) describes a classic scenario between the dentist and the dental technician. When a metal ceramic prosthesis destroys the harmony of the natural dentition, the technician often blames the dentist for improper shade taking and the dentist blames the technician for poor matching of colour. An American, O' Brien (1988:299) also noted that no two teeth are alike in either shape, colour or internal structure, and deemed recreating an acceptable replacement for natural teeth an improbable accomplishment. Therefore, from a global standpoint, there is a need to quantify the colour of the natural tooth and recreate an acceptable substitute which will be accepted by the patient, dentist and the dental technician.

2.2.3 DEMANDS FROM LOCAL INDUSTRY

Base metal alloy sales are on average higher than those of semi-precious or precious metal alloys by weight (Personal communication, Pert, 1993). This indicates that the cheaper base metal alloys are being purchased because of their strength and cost factors. However the view of respectable members of the local commercial industry (Lourenstine, 1993) is that in general, base metal alloys do not perform satisfactory in terms of colour. The area where the break in the natural harmony of the restoration and the natural dentition occurs is the cervical third. Technicians interviewed attributed the dark, unsightly colour of the free gingiva and cervical third of the restoration to the underlying colour of the base metal alloy oxide (Waddell, 1993).

2.3 OVERVIEW OF THE PROBLEMS ASSOCIATED WITH COLOURIMETRY IN THE DENTAL ENVIRONMENT

2.3.1 PROPERTIES OF COLOUR AND LIGHT

Colour is the specific sensation of perceiving different wavelengths of light (Lund, T., Schwabacher, W. and Goodkind, R., 1985:790-791). Lund and associates noted the following:

- a) that ceramo-metal restorations are produced by applying different layers of porcelain, and that the process of applying ceramic is a very complex one.
- b) that, by overlapping different colours, a different resultant colour is produced.

Lund *et.al.* (1985:790-791) reported that there are two types of colour mixing phenomena that affect the resultant colour of final restorations:

- a) additive colour; which results from the mixing of primary coloured pigments (red, green and blue) found in ceramics producing secondary colours (yellow, magenta and cyan).
- b) selective absorption (subtractive mixing) of secondary colours notably yellow, magenta and cyan from different wavelengths of reflected light.

The problem with addition or subtractive colour mixing phenomena is that dental ceramic materials pigments scatter, as well as absorb light. This basically means that both additive as well as subtractive perception is found in ceramics (Fig. 4.1).

However, in terms of objective colourimetry, the modern instrumentation does not only have to measure different types of colour mixing phenomena, as there are many other factors involved

in tooth-coloured materials, such as:

- translucency
- transparency
- fluorescence.

These factors play an important role in obscuring the subjective analysis of colour. Consequently the need for evaluation by means of instrumentation as a form objective colourimetric analysis of tooth-coloured materials has been identified (Lund *et. al.*, 1985:790-791).

2.3.2 INSTRUMENTS USED FOR THE TESTING OF TOOTH-COLOURED MATERIALS

O'Brien (1988:299-302) reported that it is inherently difficult to make an accurate measurement of tooth-coloured materials. There are many different factors that affect the accurate measurement of the colour of teeth, some of which are:

- the reflectance abilities of the crystalline structure found in natural tooth structures and in ceramics
- the differences in perception from one person to the next, i.e. subjective analysis.

It is for this reason that it is almost impossible to duplicate the exact colour of a patient's teeth without the aid of a reliable colour measuring instrument.

Various studies have been conducted to find an objective instrument which will produce repeatable and valid colour readings (Muia, 1982; Yammamoto, 1985; Goodkind, Judson and Desjardins, 1985; Lund *et. al.*, 1985). O'Brien (1988:299-302) reported that the following were required to produce a satisfactory colourimetric instrument and related computer hardware for the measurement of tooth colour readings:

a) The measuring instrument should :

- produce accurate results that are repeatable, yet have a certain amount of tolerance to handling
- produce readings that can be interpreted onto a colour coordinate system
- give results that will relate as closely as possible to what the eye sees.

b) A computer system that will:

- accept the readings produced by the instrument and match
- match readings to the restorative materials
- be user friendly and compact
- be cost effective for the average dental technician or dentist.

Two basic systems that have been used to date for the measurement of tooth-coloured materials are:

Colourimetry

A colourimeter principally measures colour in a broad band of spectrum; more specifically it measures the response of the primary colours (red, green and blue) within the spectrum. The fundamental approach to the design of this instrument was to imitate the eye and its visual responses. The only shortcoming of the colourimeter is that the instrumentation's measurement components imply that small variations in colour cannot be measured objectively, which is often the case with tooth-coloured materials.

Goodkind *et al.*, (1985:105-109) tested the relative performances between the chromascan and the spectrophotometer against those of humans, involving one hundred natural teeth. The results showed that human observers were more subjective than the spectrophotometer readings and furthermore, that the spectrophotometer produced results which were more objective than the results of the chromascan.

Spectrophotometry

The difference between the chromascan and the spectrophotometer is in the range of measurement. Spectrophotometers have been used in a number of research studies for the measurement of tooth-coloured materials (Goodkind *et al.*, 1985:105-109). Buyukyilmaze (1994:372-382) used a spectrophotometer to measure the colour stability of denture base polymers after immersion into coffee, tea and water. The results produced were easily understandable and extremely objective in nature. The common feature in all the research investigations using spectrophotometers is that the instruments have the ability to measure reflected light in terms of wavelength. A second advantage of the spectrophotometer is that it can measure colour over the entire visible spectrum.

If intermediate oxide layers of base metal alloys affect the shade of ceramics and the differences in shade between one metal-ceramic alloy and the next is not visibly noticeable, then an accurate

and objective instrument is required to carry out the reflectance testing of the specimens. Hence a spectrophotometer was selected for this investigation.

2.3.3 METHODS OF EVALUATING COLOUR IN DENTISTRY

It is an accepted fact that colourimetry is of utmost importance in dentistry (McLean 1979, 117). Due the pressures placed on the patient by society to have natural looking teeth, which include restorations that may be visible to the public, the task of matching natural teeth to prosthetic restorations, has become extremely demanding. However, most of the evaluations which are made by both dentist and technicians are highly subjective, and it takes a highly trained practitioner to have a perfectly accustomed eye for colour discrimination and perception. Many researchers have tried to find methods of perceiving colour objectively, but they have only been partly successful in their endeavours. Although this is so, a wealth of information has been gathered in terms of colourimetry and objective perception (Culpper, 1973; McLean, 1979; O' Brien, 1988; Yamamoto, 1992).

Not only has the actual method of attaining objective colourmetric readings, which are consistent and universal, been a problem, but producing a system which can clearly indicate the exact colour in terms of value, hue and chroma has also posed a few problems. One of the more frequently described systems is that of the Munsell colour order system (Lund *et. al.*, 1985:790-6). The Munsell system plots the colour on a three dimensional solid which depicts value from 0-10 (0 being black and 10 being white) and hue and chroma radiating in the form of a sphere from the centre of the solid. There have been many systems like this; however none has been as popular as the Munsell colour system. The drawback of the Munsell system is that the final analysis is subjective. Consequently, close comparisons, specifically between shades which are very close to each other in terms of hue, are difficult to grade without objective analysis. Hence the need for an objective system which will provide accurate and objective shade analysis, has become vital. Recently used systems like the chromascan and the spectrophotometer have produced results which are reasonably accurate and objective when tooth-coloured mediums are analysed.

Jacobs , Goodacre, Moore and Dykema (1987, 138-145) studied the effects of porcelain thickness and the type of metal-ceramic alloy on the colour of porcelain. Their study used two methods of

evaluating ceramic colour:

- a) Spectrophotometer data of the specimens were recorded for quantitative analysis and the C.I.E. (Commission International de l'Eclairage) notation was used and then converted to the Munsell colour system.
- b) Visual analysis of the sample groups, involving fifteen people, five of whom had anomalous colour vision and the remainder normal vision.

Jacobs *et al.* used visual examiners to ascertain whether the visual results compared well with the more objective results of the spectrophotometer data. They found that visual analysis and the spectrophotometer measurements produced similar results, but subtle changes were noted more often with the eye than with the spectrophotometer. This is an important finding since a large percentage of ceramic colourimetry is done using objective instrumentation only.

Crispin, Seghi and Globe (1991, 351-356) studied the effects of different metal-ceramic alloys on the colour of opaque and dentine porcelain. They applied shade A1 opaque to five different metal alloy groups. The opaque specimens were then measured with a chroma-meter using the CIELAB colour co-ordinates, and the differences in colour were then calculated using the CIELAB colour difference formula (ΔE). Finally the results were statistically evaluated by analysis of variance and the Turkey test. The results showed that after the opaque firing the opaque differences were insignificant; whereas after the application of the dentine layer both the palladium-silver and nickel-chromium groups showed significant differences to the rest of the metal alloys. An important fact that they noted was that it was difficult to find acceptable tolerance levels under normal viewing conditions. The findings of their investigation compared well with previous studies (Lund *et al.*, 1985).

2.3.4 PROBLEMS ASSOCIATED WITH METAL CERAMIC COLOURIMETRY

One of the greatest issues associated with manufacturing metal ceramics, is that the final restoration colour is compared to a shade guide which is made from a completely different material, notably acrylic or, in the case of the popular Vita Lumin Shade Guide, aluminous porcelain (Muia, 1982:31-32). The problem with matching shade guides to metal-ceramics is that the manufactured shade guides do not have a metal foundation. This problem was solved by introducing a custom made shade guide (Muia, 1982:32). This system has its advantages in that, if oxides from base metals do effect the shade of the final restoration, then the difference in colour can be corrected by selecting a more suitable match. Unfortunately this system is not universally

applicable to all types of metals and it would be impractical to produce custom made shade guides for every metal produced to date.

Yamamoto (1985:293-295) discussed the problems associated with dark metallic oxides in great depth and he identified three critical problem areas :

- a) The colour of metal oxides should be light, and the darker the shade of oxide the more difficult and time consuming it is to mask it out.
- b) An option to masking the dark oxide layer is to gild the metal surface with a gold surface conditioner. The problem he found when using gold surface conditioners was that the underlying gold colour affected the opaque layer, producing a highly reflective surface. However, without the gilding layer the opaque layer had to be applied very thickly in order to mask out the dark oxide.
- c) The effects of the dark oxide resulted in restorations that appeared to have translucency. By adjusting the thickness of the opaque layer the amount of translucency required could be adjusted.

When relating Yamamoto's analogies to this study, the only drawback found was that the shade used in this study was light, and required little translucency in the cervical areas.

Ideally a solution to the problems associated with shade matching would be to produce a satisfactory foundation (gilded layer) that will produce repeatable colour. As stated previously, manufacturers have produced various gilding materials which are designed to increase the bond strength and mask the effects of the dark metallic oxides produced by base metal alloys. However, metal surface conditioners are not the only available means of reducing unsightly coloured oxides. The Tanaka Sunrise system manufactured by Tanaka, Japan, introduced an alternative system which adapts a patent gold foil to the tooth preparation. The foil is manufactured in such a way that it allows the porcelain to be bonded to the gold foil. The use of a warm coloured noble metal, i.e. gold, forms the rationale behind the Sunrise System and is similar to the principle used for metal conditioners in that the objective is to produce an aesthetically pleasing coloured foundation. There are still problems when using metal conditioners for example the high reflectivity of gold. However, when applying gold foundations (whether the foil or application paste method is used) a basic increase in warmth in the colour of the restoration is achieved.

2.3.5 PORCELAIN AND ITS EFFECTS ON SHADE

According to (Muia, 1982:96), the most important ingredient of the entire ceramic restoration is the opaque layer. Poor opaque layering is the main cause of colour failure and the largest contributor to a lifeless looking crown; yet it is the area given the least attention and effort by ceramists.

Dental porcelain is normally coloured by fritting high temperature resistant pigments. These pigments are normally metallic oxides and are found in most opaques (McLean, 1980:56), for example:

- chromium oxides
- cobalt salts.

Chromium oxides produce a green colouring and cobalt salts are used to increase the blue colouring of enamels as well as the graying of dentine. In certain shades iron oxides are used instead of cobalt salts. All these oxides are present in ceramics to a certain degree. McLean (1980:56-57) reports that technicians complained that some porcelains assumed a greenish hue after firing, and that the inherent greenness could be accentuated by over firing. He offers two possible explanations to the greenish hue that the technicians observed:

- a) The colour of the inherent oxide pigments of the porcelain was accentuated by the addition of new base metal oxides produced at the metal surface. These base metal oxides were probably of the same colour as the oxide pigments found in the porcelain.
- b) The boundary of the porcelain crystal lattice dissolved due to over firing and consequently a highly translucent green coloured restoration was observed.

What McLean does not indicate, is whether precious or base metal alloys were used. This potential problem can be compounded by chromium and cobalt oxides produced from the metal substructure interface of base metal alloys. Crispin *et al.* (1991:356) noted that there was a decrease in the luminance reflectance factor of the nickel sample group used in their study, and assumed that this phenomenon was due to nickel oxides affecting the resultant colour. They did not, however, confirm this fact with microscopic viewing, as this was not the area in which the study was involved.

Therefore, hypothetically, the opaque should not be affected by base metal alloys which produce dark oxides, whether green or black. Metals which produce relatively lighter coloured oxides, for example the beryllium containing nickel-chrome alloys, should be used (Phillips, 1982:548). It has been reported that the local dental laboratories tend to avoid base metal alloys that have beryllium due to the toxicity of the element (Pert, 1993).

Jacobs *et al.* (1987:137) studied the effects varying thicknesses of porcelain have on the shades of final restorations. They noted that there were significant differences in the shade of dentine when different thicknesses of porcelain were used (0.5mm, 1mm and 1.5mm). An important finding was that when the opaque porcelain colour more closely matched the dentine colour, as in the case of the B shades, a colour match could be achieved with less overall porcelain thickness.

2.4 METAL CERAMIC ALLOYS USED IN DENTISTRY

2.4.1 BASE METAL ALLOYS VERSUS PRECIOUS METAL ALLOYS

One of the greatest advantages of using non-precious metal ceramic frameworks as opposed to the softer precious metal, is the decrease in thickness of the metal substructure without loss of strength (Phillips, 1982:555). This characteristic is highly advantageous with restorations that have limited space for ceramics on the labial sections of the restoration. The modulus of elasticity of the base metals is approximately twice that of the precious metal alloys, which enables the technician to manufacture long-span bridges without having to concern himself with too much flexibility and fracture of the brittle ceramic.

There are many factors that militate against the use of non-precious metals, although their physical characteristics portray non-precious metals as being the most suitable metal for metal ceramics. Derman, Groll and Kump (1992:156) noted that the corrosion resistance of precious metal was higher than that of most base metal alloys due to their high electrochemical potential. In practice this means that the precious metals are highly stable when in a hydrogen saturated environment, such as in the mouth of a patient. Not only are the precious metals biocompatible in terms of toxicity, but they also offer a better marginal seal due to their burnishing ability.

The literature continually describes troubleshooting associated with masking out the unsightly dark oxide colours produced by base metal alloys (Crispen *et al.*, 1991:356). In the commercial

environment dental technicians have the following complaints about base metal oxides:

- The base metal oxide is always visible when using specifically the lighter shades (e.g. VITA A1, B1).
- By increasing the opaque layer the result is a reflective surface which is too light.
- The dark oxide is especially evident in the cervical areas where the ceramic is thin.

The demand from the commercial environment has always been for a dentine (natural tooth-coloured) coloured foundation which requires little masking in terms of the thickness of opaque. The obvious solution is the use of a gold coloured metal-ceramic alloy or a surface conditioner which produces a golden hue. Industries' response was the introduction of economic surface conditioners and certain gold foils which could be used with ceramics. The result was the introduction of some of the following surface conditioners:

- Deck-Gold (Degussa, AG)
- Spectra Seal (UNITEK, California)
- Vita Spectra-Gold (Vita, Germany).

2.4.2 BASE METAL OXIDES

Yamamoto (1985: 108-120,139) reported that the colour of the oxides produced is dependent on the metal used. He found that most of the metal-ceramic base metals produced a dark unsightly oxide, as opposed to the thin, golden brown coloured oxide which was produced by the precious metals. Ideally, the oxide layer is white with a natural golden yellow hue; however, most of the base metals used to enhance physical properties in metal-ceramic alloys produce a dark oxide colour. According to Yamamoto, the base metal elements in precious and semi-precious alloys Sn, In, and Fe produced white, gray, and red metal oxides respectively when a low vacuum firing cycle was used. Yamamoto also noted that the colour of the produced oxides could be selectively removed by acid pickling the metal surface, whereby the unwanted oxides like Fe and In are removed and the light coloured Stannic oxides left behind. Most opaque porcelains are designed to mask these colours. However, the oxide colours produced by base metal alloys are usually dark green and most opaques have difficulty masking out this colour.

The most commonly found base metal alloys to date are categorized into two main groups :

- a) nickel-chrome
- b) cobalt-chrome.

The differences between the variations of metals are in their compositions. A standard nickel-chrome and cobalt-chrome alloy normally have the following basic compositions (Tuccillo, 1993:366):

<u>Nickel-chrome</u>	<u>Cobalt-chrome</u>
77% Ni	67% Co
13% Cr	21% Cr
4% Mo	8% Mo
6% Al	4% Al
(2% Be)	(3% Ga,Ru)

(Note: Elements in brackets indicate the possible inclusion or exclusion of these elements in the composition of the base metal alloy.)

Cobalt-chrome can be further categorized into whether the metal alloy contains ruthenium or not. The presence of these minor base metal elements has an effect on the base metal alloy oxide colour. The effects of ruthenium, however, are not as significant as the effects of beryllium in nickel-chrome containing alloys. The reason for the interest in beryllium containing alloys is that beryllium has an effect on the colouring and amount of oxide produced by nickel-chrome alloys. It was therefore expected that the results in this study would support the use of beryllium-containing alloys despite the fact that beryllium is a toxic element.

The most popular alloys are the nickel-chromium type (McLean, 1980:33). The reasons for its popularity are its corrosion resistance and strength, as well as its resistance to creep. However, Dudek (1988:85-86) states that cost is the major factor in popularity of these alloys, despite their poor colour and the associated problems of masking their dark oxide layer.

Unfortunately, metals such as chromium or nickel present considerable problems during metal-ceramic firing. McLean (1980:33) states that the following critical factors occur when a

chromium or nickel element is present during oxidation:

- a) A layer of nickel and /or chromium oxide rapidly builds up at the ceramo-metal interface.
- b) Depending on the preparation of the metal surface and the length of the firing cycle, the oxide will vary in thickness. Longer firing cycles will inevitably produce thicker oxide layers.
- c) The colour of the oxide layer is dependent on:
 - the amount of oxide produced on the metal surface
 - which base metal element is reduced to the surface.

The resultant oxide which is reduced to the metal surface during the firing cycle, will affect the colour of the opaque and thereby influence the thickness of opaque required to mask out the dark oxide colour. This will consequently pose problems when there is limited space available in the cervical regions of restorations or when lighter shades of ceramic are used .

Jacobs *et al.* (1987:144) also tested three different types of metal alloy, namely:

- nickel-chromium
- high palladium alloys
- gold-platinum-palladium.

Jacobs *et al.* (1987:144) found that there were significant differences between the hues of the nickel-chromium group and the high-palladium and gold-platinum-palladium groups. The results showed that the nickel-chromium group produced a darker shade than the high-palladium and gold-platinum-palladium groups. Crispin *et al.* (1991:356) did a similar investigation although they included a palladium-silver group (palladium-silver is well known in the commercial environment as having a disproportionate oxide layer when compared to other metal-ceramic alloys). They noted that the palladium-silver and nickel-chromium alloy groups caused significantly greater colour changes relative to the high-gold-containing control at the body porcelain stage. However, Crispin *et al.* (1991:356) improved on the study by Jacobs *et al.* (1987) in that their study highlighted the fact that no significant colour changes were noted with alloys for the opaque stages for all alloys.

2.4.3 THE GILDING SOLUTION TO METAL-CERAMICS

In terms of base metal alloys, manufacturers have accepted that adequate opaqueing of the metal surface will provide sufficient masking of the dark oxide layer. Yet manufacturers have

introduced gilding materials to mask out the effects of dark oxides colours produced by semi-precious metals. However, when the oxides of a semi-precious metal and a base metal alloy are compared visually, there is not much difference in terms of colour.

Vita (AG, Germany) introduced a metal conditioner which produces a satisfactory bond between both semi-precious metal alloys and ceramics. The advantages according to Vita are the following:

- a) The metal conditioner (gilding material) is manufactured from gold, which is noble and non-toxic.
- b) The gold coloured surface boasts a yellower (natural dentine colour) coloured foundation for ceramics, which in turn will produce a warmer coloured final restoration which is closer to the colour of the natural tooth.
- c) The gold surface increases the bond strength between the metal alloy and the porcelain.

Different manufacturers have introduced a variety of gilding materials (Vita, Spectra-Gold paste; Unitek, Spectra Seal; Degussa, Deck-gold) and each promotes a new concept. However, most of the gilding materials, whether in a mixing kit or simply in the form of an application paste, are based on the same principle.

Since manufacturers have spent time producing a solution to mask out the dark oxide layer of semi-precious metals, a debate has been opened as to why a similar development for base metal alloys has not taken place. A hypothetical argument in support of the manufacturers might be based on the following:

- Gilding materials do not generally bond to base metal alloys, with the possible exception of Blendgold Neu (Heraeus Edelmetalle, Germany) which seems to be ever increasingly technique sensitive.
- The opaques manufactured by the industry do in fact mask out the dark oxide layer of base metal alloys.
- Semi-precious metal alloy oxides require a more stringent method of masking the dark oxide, due to an inherent composition flaw found in semi-precious metal alloy oxides.

- Because semi-precious metal alloys have better physical properties than the base metal alloys and are less technique sensitive in the laboratory, it may be better to use a more expensive metal and achieve better results.

There are obviously two sides to the debate as to why the base metal alloys do not have surface conditioners. It is important to clarify the difference between bonding agents and gilding paste in terms of colour. In the past many commercial technicians have purchased surface conditioners or bonding agents in the hope that they may achieve better results, not only in terms of bond strength but also to enhance the aesthetic appearance of a restoration. The results of such purchases have left the commercial technician with a lack of confidence in the success of these so-called 'gilding materials'. It is imperative that the reader understands that bonding agents are applied to increase the metal-ceramic bond, and gilding pastes are normally applied to increase the yellow hue of the metal alloy foundation. Although manufacturers have managed to combine gilding materials and bonding agents, the gilding paste, which was investigated in this study, only serves as an aesthetic enhancer.

In the light of the above discussion, it was argued that there was a need to find out whether gilded semi-precious metal alloys would produce better properties of colour than opaqued base metal alloys. It was also necessary to determine how each group of alloys would compare with the shade guide.

2.5 SUMMARY

The foregoing literature review is summarized as follows:

- Due to the increase in the price of gold, base metal alloys are being introduced into the commercial market in increasing quantities. A second reason for the increase in their popularity is that precious metal alloys lack the strength and stiffness that base metal alloys boast.
- Base metal alloy oxides are dark in colour, as opposed to precious metal alloy oxides which display light coloured oxides. The dark oxides found on base metal alloy surfaces are attributed to the presence of base metal elements such as chrome, iron and cobalt in the alloy's composition. There are two basic formulations of base metal alloys; nickel-chrome and cobalt chrome. It has been reported that minor elements such as beryllium and ruthenium within base metal alloys can affect the colour of the base metal oxide.

- The problem with metal-ceramic colourimetry is that the shade guide which is used to match the patient's tooth colour to artificial tooth colour materials is made out of a different materials which is different from the porcelain used for restorations. Oxide pigments found in porcelain can combine with oxides found on the metal surface affecting the overall shade of the restoration. Manufacturers have produced gilding material to cover the effects of the dark oxide found in semi-precious metal alloys.
- Previous studies have tested the effects of varying porcelain thicknesses and found that by reducing the thickness of the body porcelain the colour of the restoration will become darker .
- The colour of metal-ceramic restorations at the opaque stage should be similar for all metal alloy groups.
- Nickel-chromium and palladium alloys produce darker coloured restorations than the high-gold metal alloys.
- Previous methods of colourimetric testing have shown that the chromascan readings are subjective as opposed to the objective analysis of spectrophotometers.
- Manufacturers claim that by gilding a semi-precious metal alloy, the result will provide a close approximation to the natural colour of the shade guide and natural teeth.
- It is evident that no recent studies have established the effects of different base metal alloys' (Ni - Cr, Co - Cr, Ni - Cr [Beryllium]) oxide colours, and the deleterious effects that these dark oxide colours have on the lighter shades of ceramic e.g. A1, C1 Vita shades. It was primarily the latter observation that prompted this study.

CHAPTER THREE

METHOD

3.1 THE DATA

3.1.1 PRIMARY DATA

The following primary data were obtained to address subproblems one, two and three (section 1.2.2):

- The mean spectral reflectance values and tristimulus values of the five selected base metal oxide layers and the gilded layer of the control group.
- The mean spectral reflectance values of all six metal alloy samples, after applying three ceramic layers and one simulated glaze firing to each specimen.
- The mean spectral reflectance values of the manufactured shade guide.
- The statistical values obtained from the two-sample analysis of all the sample groups.

3.1.2 SECONDARY DATA

The following secondary data were obtained to address subproblems two and three:

- The DIN 5033 (1964) Chromaticity diagram which was used to display the tristimulus values of the different ceramic layers.

3.1.3 THE CRITERIA GOVERNING THE ADMISSIBILITY OF THE DATA

- Only the data and statistical analyses recorded on the Texflash Reflectance Spectrophotometer (Datacolour-Colour future, U.S.A) at Romatex Textiles, Jacobs Moberi, Durban were used.
- Only the metal-ceramic specimens made by the researcher in the dental laboratory of Technikon Natal were used.

3.2 METHODOLOGY

The experimental method was used in this investigation.

The first step was to identify the most popular metal-ceramic alloys. The reason for surveying commonly used brands was to ensure that the results would have applicational value in the commercial dental industry when published (Table 3.1).

The second step was to establish the effects of the oxide colour on the shade of the porcelain. Thirty specimens for each of the five selected base metal alloy groups were fabricated using the lost wax casting method. The total number of base metal alloy specimens was 150. A further 30 specimens were cast in a semi-precious metal alloy to provide a control group. Each specimen was marked to allow individual recognition. Following this all the base metal alloy sample groups were subjected to an oxide firing, whereas the surface of each specimen in the control group was gilded with a gold layer. At this stage the base metal alloy groups and the gilded control group were tested using a spectrophotometer (Texflash - spectrophotometer for reflectance measurements). A measured porcelain layer was then applied to the metal surface of each specimen, and the appropriate firing cycle (Table 3.3) was used to fire the porcelain. Subsequently the colour of the porcelain of each alloy group was recorded using a spectrophotometer. This process was then repeated until a simulated ideal metal-ceramic crown was built up and fired.

In order to relate the control group to the shade guide, a custom shade guide was manufactured. The manufacturing of the custom shade guide was necessary due to the fact that shade guides used in industry were not suitable in shape and size for measurement in the spectrophotometer aperture. The shade guide was then tested in the same manner as the rest of the experimental groups.

The data recorded from the spectrophotometer were subjected to statistical analysis by means of the software installed in the computer, which was directly linked to the spectrophotometer. By comparison the mean spectral reflectance data obtained from the spectrophotometer produced results that showed which base metal alloy displayed a similar spectral reflectance to the gilded control group and the custom made shade guide. This in turn showed which metal-ceramic base metal alloy produced the most desirable oxide layer.

3.3 SAMPLE SIZE

The central limit theorem states that for sample sizes greater than thirty the sampling distribution of the mean has a normal distribution and therefore a sample that is equal to or greater than thirty is recommended for the purpose of valid statistical analysis. Therefore a sample size of thirty was used for each experimental group.

3.4 GENERAL DESCRIPTION OF THE MATERIALS AND METHODS

3.4.1 INTRODUCTION

The ensuing section provides a detailed description of the materials and the methods used to conduct this study. It must be emphasized that the experimental nature of this investigation should allow subsequent researchers to duplicate the methods accurately producing similar results. It should be noted however, that any deviation from the subsequent methods may affect the data and hence the results. A detailed discussion as to why certain materials and methods were used is offered in section 3.5. This will enable the reader to have full understanding of the entire methodology of the study.

3.4.2 THE TELEPHONIC SURVEY

The first step was to carry out a telephonic survey of the commercial brand names of the base metals most commonly used in South Africa. From this survey, five brands of metal-ceramic base metal alloys and one brand of semi-precious metal alloy for the control group were selected. The reason for surveying commonly used brands was to ensure that the published results would have applicational value in the commercial dental industry.

The six brands chosen are listed in Table 3.1

Table 3.1: Telephonic Survey

BRAND NAME OF ALLOY	MINIMUM PURCHASE WEIGHT (g)	PRICE/ g quoted on 2/2/1993	AVAILABLE ?	SELECTED?	REASON FOR SELECTION
Ducranium	200g	R 0.54/g	✓	✗	failed random selection
Sancan	1000g	R 1.32/g	✗	✗	not available
Remanium CD	250g	R0.54/g	✓	✓	price, nickel-chrome
Ceramco	100g	R0.55/g	✓	✗	failed random selection
Rexillium	142g	R1.69/g	✗	✗	too toxic
Wiron 99	SQ*	R1.69/g	✓	✓	very popular
Bond-on-4	SQ*	R24.60/g	✓	✓	popular
Pors-on-4	SQ*	R24.60/g	✓	✗	not as popular as Bond-on-4
Ceradium V	250g	R1.69/g	✓	✓	popular, includes beryllium
Wirobond	250g	R0.54/g	✓	✓	popular, cobalt-chrome
Heranium	100g	R0.54/g	✓	✓	random selection

(* available on request)

3.4.2.1 SELECTION OF BASE METAL ALLOYS

The selection of the metal alloys used in this study was based on their availability, price and their physical compositions . Their popularity was also taken into account in terms of sales from the suppliers. The remainder of the metals that were not selected for any specific reason, were randomly selected. Therefore the following metal alloys

were selected:

BASE METAL ALLOYS

- REMANIUM CD (nickel-chrome)
- CERADIUM V (nickel-chrome, beryllium)
- HERANIUM (nickel-chrome)
- WIROBOND (cobalt-chrome) (beryllium and nickel free)
- WIRON 99 (nickel-chrome)

As noted previously the base metals were selected in terms of their popularity, cost and availability. There were, however, a few exceptions to the stated criteria. Although Ceradium V contains a certain percentage of beryllium, which is a toxic element, it was selected to represent the beryllium-containing base metal alloys in the study. Wirobond on the other hand is non-toxic and was selected to represent the cobalt-chrome base metal group. Moreover, it was widely popular in South Africa at the time of the survey.

SEMI-PRECIOUS METAL ALLOYS

- BOND-ON-4

Due to its popularity, Bond-on-4 was selected to be the control group metal alloy over Pors-on-4. It should be noted that Bond-on-4 is a semi-precious metal alloy and it was used because a precious metal gilding layer was to be fired over the metal alloy to promote a yellow, dentine coloured foundation, onto which the ceramic was to be fired. The gilded layer was applied in an attempt to mask out any effects of an underlying oxide layer. Therefore the effects of the semi-precious metal alloy oxide were irrelevant in the study and the metal alloy should be recognized only as the control group.

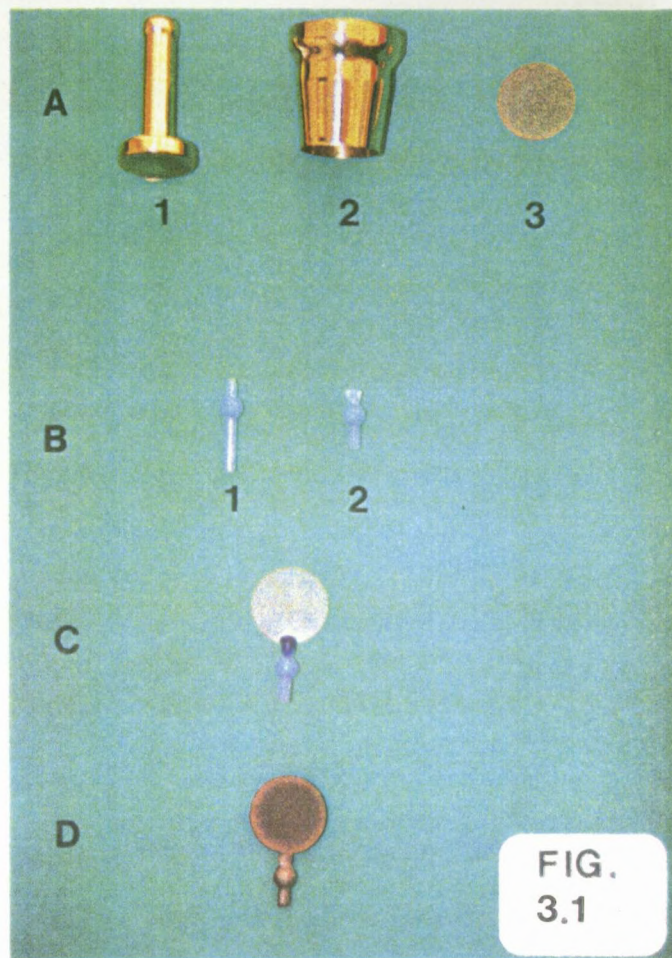


FIGURE 3.1: SEQUENCE OF TEST SPECIMEN PREPARATION

A1 - Pattern plunger

A2 - Cylindrical cutting tube

A3 - Wax pattern

B1 - Di-equi hollow reservoir sprue pins

B2 - Sprue pin reduced to half with attachment point flattened

C - Wax pattern attached to sprue pin

D - Cast specimen

3.4.3 WAX PATTERN PRODUCTION

3.4.3.1 WAX PATTERN PRODUCTION

In order to establish the effects of the oxide colour on the shade of the porcelain, 30 specimens for each of the sample groups were fabricated (30 x 5 sample groups + 30 control group = 180 specimens in total). Thirty control group specimens were also fabricated using the same process as for the base metal alloy sample groups. The specimens were manufactured using the lost wax casting method.

The wax specimens were punched out of a 0.4mm Kerr casting wax sheet (Millner's Dental Suppliers, Cape Town, South Africa). The specimens were then carefully removed from the finger punch described in section 3.5.2 (Fig. 3.1) by means of the pattern plunger. The patterns were then placed onto a flat surface so as to ensure that the thin patterns remained distortion free.

3.4.3.2 SPRUING OF WAX PATTERN

Di-Equi Hollow Reservoir Sprue Pins (8-10 gauge) (EZ Casting System, Wappingers Falls, U.S.A) were selected as suitable spruing pins (Fig. 3.1 [B1]). These specific spruing pins were selected due to the plastic being soft in nature, facilitating an uncomplicated burn out process during the burn out cycle .

The length of each sprue pin used for spruing was reduced to half by cutting the sprue with a scalpel blade to ensure that, after the sprue pin and wax pattern had been attached, they fitted into the 4x muffle former (Degussa AG, Germany). The attachment point of the sprue pin was also flattened with a flat nose orthodontic pliers to enable easy attachment to the wax pattern and a more suitable metal delivery to the pattern during casting of the specimens. The adjusted sprue pins were then attached to the outer circumference of the wax patterns (Fig. 3.1 [C]). This was done by welding the two parts together with blue inlay wax (Degussa AG, Germany) using a Hot Rod Powered Wax Knife (Natal Technikon, South Africa).

Ten sprued patterns were then attached to a muffle base with blue inlay wax. Each pattern was aligned parallel to the next so that all ten specimens would fit into the muffle. The

specimens were positioned in such a manner that a 3 degree taper straight cut burr could move freely between two specimens to facilitate cleaning.

3.4.4 CASTING OF WAX SPECIMENS

3.4.4.1 CALCULATING THE AMOUNT OF ALLOY REQUIRED FOR CASTING

To facilitate easier working space during the recovery and cleaning stages described in section 3.4.5, batches of ten wax patterns were attached to a 4X sprue former (Degussa AG, Germany). The amount of metal alloy required was calculated in the following way (Waddell, 1993:33):

$$A \times B = C$$

A= weight of sprue former with the attached patterns
minus the weight of the sprue former.

B= specific gravity of the alloy.

C= weight in grams of the alloy required to cast the
pattern.

Each sprue former was weighed and the weight marked on the underside of the muffle former to assist in the calibration of the actual weight of the wax patterns.

Having calculated the weight of metal alloy required to cast one single muffle, a single ingot of metal alloy was weighed. The total weight required to cast the ten specimens (C) was then divided by the mass of the single ingot (D). The result would be the number of ingots required for casting (E).

$$C/D = E \text{ (number of ingots)}$$

The number of ingots (E) was rounded off to the nearest round figure. At this stage, however, the number of ingots was only sufficient to cast the wax pattern and the spruing channels. This allowed no margin for error, therefore an additional ingot was added to the total number of ingots to ensure that the casting was successful. The total number of ingots inclusive of the additional ingot was marked on the muffle former base.

3.4.4.2 INVESTING OF WAX PATTERNS

Three muffles were invested at a time, which represented one base metal alloy group of thirty specimens. This procedure was chosen to avoid investment setting during the pouring of the molds. Because the specimens were not required to fit any preparation or die, there was no reason to mix any expansion liquids with the investment. The purpose of the mold was to simply reproduce the wax patterns in the desired base metal alloy.

A single Kera-Vlies muffle ring liner 1.0 X 50mm (Denturum, Germany) was soaked in water as per the manufacturer's recommendation, and placed inside the muffle. As stated previously, expansion was not required, therefore the only purpose of the single ring liner was to aid in divesting the cast specimens. Before placing the muffle former over the muffle former base, the wax patterns were sprayed with a wetting agent, Wax-it spray (Degussa A.G, Frankfurt). The muffle former was then placed carefully into position inside the muffle former base.

Four 60g packets of Bellavest T (Bego, West Germany) were placed in a Miltivac 4 Mixing bowl (Degussa A.G, Frankfurt) and mixed with 56ml of distilled water. The Bellavest T and liquid was spatulated for about 10-15 seconds until the mass was well wetted and free from lumps. The investment was then mixed in the Miltivac 4 mixing machine (Degussa A.G, Frankfurt) for 60 seconds.

The investment was then poured into the muffle and allowed to set for one hour. The rubber muffle bases were then removed and were ready to be placed in the burn out furnace.

3.4.4.3 BURN OUT CYCLE OF FURNACE

All burn out cycles were conducted on a (KWO/EWL Type 5645, Germany) burn out furnace. The muffles were then placed in the burn out furnace, and the following staged burn out was used:

1. Room temperature to 250°C (60 min.)
2. 250°C to 650°C (40 min.)
3. 650°C to 950°C (60 min.)

NOTE:

- The times indicated are the temperature holding times. The burn out cycle for Bond-on-4 is the same as the above, however, the top heat soaking temperature was reduced to 850°C to compensate for the lower melting temperature.
- Only three muffles were placed into the burn out furnace at a time. This was done to ensure that each specimen of any one base metal alloy group had the same burn out cycle.

3.4.4.4 INDUCTION CASTING OF SPECIMENS

Once the muffles had reached 950°C the ceramic crucibles were placed in the furnace for five minutes to reduce the thermal shock of the smelting process.

The induction casting machine (Galloni 3N Modular, Italy) was switched on five minutes prior to the casting process to allow for the warming of the electrical components (as recommended by the manufacturer). The casting machine settings were as follows:

- Magnetic flux was set at **1500 Hz**
- The electronic 'eye', was set on **stabilize**

The crucible setting was switched to **2 ceramic**

The crucible was then removed from the furnace and placed in the crucible cradle of the induction casting machine and secured in place. The specific amount of metal (section 3.4.4.1) was placed in the crucible at which stage the heating commenced automatically. As soon as the electronic 'eye' started 'clicking' (signifying the automatic heat stabilization of the metal), the stabilizing switch was moved to the **free** position.

The muffle was then removed from the furnace and placed in the casting cradle. As soon as the metal was completely molten and there was no visible form of any remaining solid metal ingots, the casting mechanism was released.

3.4.5 RECOVERY AND CLEANING OF CASTINGS

3.4.5.1 RECOVERY OF CASTINGS

After the casting process had been completed, the muffles were allowed to bench cool until the muffle temperature was down to room temperature.

Using a Renfert Riveting Hammer No 1226-0000 (Renfert, Hilzingen, Germany) the muffle was lightly tapped until the mold broke. Any excess investment was removed with plaster pliers (Renfert, Germany) by gripping the sprue button with the pliers and lightly tapping the spruing button.

3.4.5.2 TRIMMING AND SANDBLASTING

The specimens were then sandblasted with a Topstar 2 sandblaster (Bego, West Germany) and 50 micron aluminiumoxide sand. The blasting pressure was set at 4 bar (4000 psi).

In order to remove any remaining investment or possible metal contamination produced during the casting process, the specimens were lightly skimmed with a straight cut tapered carbide burr (Nova Dental Suppliers Pty LTD, South Africa) until bare metal was showing. The purpose of using the straight cut burr was demonstrated by Yamamoto (1985:124). He found that the result of using the straight cut, as opposed to the diamond, silica or cross cut carbide burs, produced a highly superior finish which was free from any contamination from the burr itself. Each specimen was then carefully separated from its spruing channel using 25mm x 0.5mm separating disks (Bego, West Germany). The specimens were then sandblasted once again with 50 micron aluminiumoxide sand and cleaned in an ultrasonic cleaner.

3.4.6 PREPARATION OF BASE METAL ALLOY SURFACES

3.4.6.1 OXIDATION OF METAL ALLOYS

It is suggested by a certain school of thought that an oxide firing is imperative due to the increase in bond strength. Yamamoto (1985:118) demonstrated how oxide firing produces an oxide film over the metal surface before the application of ceramic; this in turn

increases the wetting of the metal surface and increases the bond strength. This is in contrast to the manufacturers' recommended surface preparation procedures for most of the selected base metal alloys in Table 3.1.

Since colourmetric evaluation of the oxide colours was the purpose of this investigation, an oxide firing was done. Furthermore, the oxide firing was done in such a manner that each individual oxide colour of the five base metal alloys could be ascertained.

All firing of oxide layers and ceramic layers was completed with a Vita Vacumat 300 (VITA, Germany). Only five specimens were fired at one time (due to the physical constraints of the firing chamber). The following firing cycle was used :

1. programme : Oxidation firing
2. drying time : 600°C for 3 min.
3. temp. climb : 55°C/min.
4. top temp. : 940°C (1 min.)
5. vacuum : none

After the oxide firing, the specimens were removed immediately from the firing tray and allowed to cool to room temperature. Following this the specimens were placed into their protective boxes. It should be noted that special care was taken to ensure that the specimens were not touched by hand. Failure to comply with this could affect the eventual colour of the oxide layer and contaminate the ceramic.

3.4.7 GILDING OF THE CONTROL GROUP

3.4.7.1 FIRING OF THE GILDING MATERIAL

Five specimens of the control group were placed on the firing tray and allowed to dry for five minutes under an open furnace (the furnace was set on stand-by temperature of 600°C). This pre-drying time was monitored manually to ensure that all liquid residue was burnt off satisfactorily. Following this, the programme was then started. The firing temperatures were programmed into the miscellaneous section of the furnace

programme, since there was no pre-programmed cycle. The firing cycle was as follows:

- pre-drying time : 5-6 min.
- climb temperature : 55°C/min.
- top temperature : 820°C
- top temperature time : 1 min.
- vacuum : none
- cooling : none

The specimens were then removed from the firing chamber immediately after completion of the programme and allowed to cool to room temperature. Following the firing of the control group, all the sample groups as well as the control group were taken to Romatex Textiles (Mobeni, South Africa) to be tested for their oxide colours.

3.4.7.2 APPLICATION OF THE GILDING PASTE

Of the top three leading brands of gilding/bonding materials available in South Africa, only one was selected for the gilding of Bond-on-4. An explanation for this choice is presented in section 3.5.5.2.

As discussed in section 3.5.5.2 Spectra-Gold paste (Vita, Germany) was the selected gilding material used in this study. It should be noted that the manufacturers recommended that two layers of material be painted on the metal alloy to produce an effective difference between bonding and colour. However, due to the cost of the material and the large surface area of each specimen, only one layer was applied. The results are shown in Figures 3.9 and Figures 3.10 (the specimen marked X).

A small drop of Spectra-Gold was squeezed from the syringe and placed in the centre of each specimen. The brush, which was supplied in the Spectra seal kit, proved to be quite adequate for the application of the paste. The paste was then painted on in one single direction until the entire area was covered with material. Following this, the paste was smoothed out by changing the direction of the brush strokes 90 degrees to the original brush stroke. This process ensured that the surface was evenly covered with material.

3.4.8 NUMBERING AND IDENTIFICATION OF SPECIMENS

The numbering method of the specimens (Table 3.2) served two purposes:

- the specimens were identifiable during the firing and testing stages
- a set of data was recorded for each specimen by the spectrophotometer and was printed out in hard copy. These sets of data were given numbers which related to the individual specimens.

The first method of identification was the marking of the specimens on the underside with opaque ceramic material. Using the opaque material ensured that the marking did not fade or burn off during the firing stages of the firing cycles. The second method of identification was to simply use the computer connected to the spectrophotometer and type the corresponding specimen number against the specimens' measurements, for example (marked specimen number 2A0PSd).

Spectrophotometer print out

Code name : Ceradium V

Sample name : dentine firing

Number : #2

Table 3.2 outlines the method used to number the sample groups.

Table 3.2: Identification And Numbering Of Specimens

	CERADIUM	WIRON 99	WIROBOND	HERANIUM	REMAIMIUM CD	BOND-ON-4 CONTROL GROUP
SAMPLE GROUP NAME	A	B	C	D	E	X
OXIDE FIRING(O)	(1-30)AO	BO	CO	DO	EO	XO
1ST OPAQUE(P)	(1-30)AOP	BOP	COP	DOP	EOP	XOGP
2ND OPAQUE(S)	(1-30) AOPS	BOPS	COPS	DOPS	EOPS	XOGPS
DENTINE FIRING	(1-30) AOPSD	BOPSD	COPSD	DOPSD	EOPSD	XOGPSD
GLAZE FIRING	(1-30) AOPSDi	BOPSDi	COPSDi	DOPSDi	EOPSDi	XOGPSDi

Note: all specimens were numbered between 1 and 30 in each sample group, and were also allocated an alphabetical number which signalled the exact stage the specimen was at any one time during the ceramic firings.

3.4.8.1 READING TABLE 3.2

Each sample group was assigned a letter of the alphabet to identify the metal alloy type. For example: Ceradium was given the first letter **A**. After each consecutive layering of ceramic a further letter was applied to the reverse surface. All the letters that were painted on the reverse surface of the specimens corresponded to the specific layer that was being applied to the specimen (Ceradium's Opaque layer had the letter **O** painted on the reverse surface). The only exception to this process was the gilding layer of Bond-on-4 (the control group).

Bond-on-4 had a gilding layer applied between the oxide firing and the first opaque layer. This layer is represented in Table 3.2 by the letter **G**. Following the gilding layer, the

numbering of the control group continued in the same manner as the numbering of the base metal alloy sample groups.

3.4.9 LAYERING AND FIRING OF CERAMIC

3.4.9.1 CERAMIC FIRING CYCLES

As stated previously in this chapter, all firing cycles were completed using a Vita Vacumat 300 (Vita, Zahanfabrik, Germany). The firing cycles were in accordance with the manufacturers' recommendations (Table 3.3). However, certain drying temperatures were adjusted. The reason for these adjustments was that five specimens were fired during one cycle, as opposed to the normal single specimen. Therefore the firing cycles were adjusted to simulate a five unit bridge (five specimens).

Table 3.3: Ceramic Firing Cycles

CERAMIC LAYER	PROGRAMME SELECTION	PRE-DRY TEMP (°C)	PRE-DRY TIME (MIN.)	TEMP CLIMB (°C/MIN.)	TOP TEMP (°C)	TOP TIME (MIN.)	VACUUM (0-100%)
OXIDATION	OXIDATIO 1	550°C	3 MIN.	110°C/MIN.	940°C	0 MIN.	0%
1ST OPAQUE	OPAQUE 1	600°C	6 MIN.	85°C/MIN.	930°C	1 MIN.	100%
2ND OPAQUE	OPAQUE 2	600°C	6 MIN.	85°C/MIN.	920°C	1 MIN.	100%
DENTINE	DENTINE 1	600°C	6 MIN.	55°C/MIN.	920°C	1 MIN.	100%
GLAZE	DENTINE 2	500°C	3 MIN.	110°C/MIN.	940°C	0 MIN.	0%

3.4.9.2 SPRAYING OF THE OPAQUE LAYER

To achieve optimum coverage of opaques ceramic, it was decided to use the spray-on technique of applying opaque as opposed to the brush-on technique. The choice of this method is explained in section 3.5.5.1.

The air pressure for the spraying of the opaque layers was supplied by a De Vilbiss compressor (De Vilbiss Co LTD, Bournemouth, England). This compressor was specifically designed for the use of air brush spraying, therefore regulation of air pressure

was not necessary (40psi). The spray gun used for the spraying technique was a Paashe Type H (Paashe, U.S.A) pencil spray gun.

The mixing ratio of Formula D-1 Specrafire (J.D Emmons, Ohio U.S.A) spraying liquid to Duceram (Ducera, W. Germany) ceramic opaque powder was 1.5g to 1ml of liquid. Due to the nature of the spraying liquid, which is designed for quick drying, evaporation may take place after spraying for long periods. To maintain spraying consistency small quantities of liquid were added.

Before spraying the specimens, the air demand valve was adjusted for maximum coverage and consistency. The spraying of the specimens was completed in a zig-zag fashion, releasing the air pressure demand button after passing over the specimen. This ensured that there was no excess build up of opaque material in isolated areas. To avoid wastage, a 2mm plastic sheet was positioned behind the specimen during spraying; the excess ceramic was later collected and re-used. After spraying in one direction, the specimen was rotated 90° to the first spraying direction, and re-sprayed in a similar fashion. The above process was considered to deliver one layer of opaque. The first opaque layer had one layer applied to the specimen surface and the second opaque layer had two applications of ceramic. Each layer of opaque was then fired as set out in Table 3.3.

3.4.9.3 THE LAYERING OF DENTINE

A detailed description of the layering depth gauge and its use is provided in section 3.5.3 . It is important that this section be fully understood before reading the methodology of the dentine layering.



FIG.
3.2



FIG.
3.3



FIG.
3.4

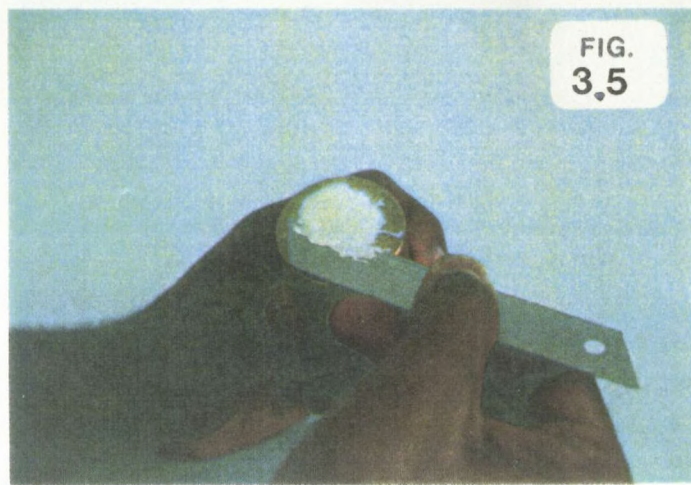


FIG.
3.5



FIG.
3.6

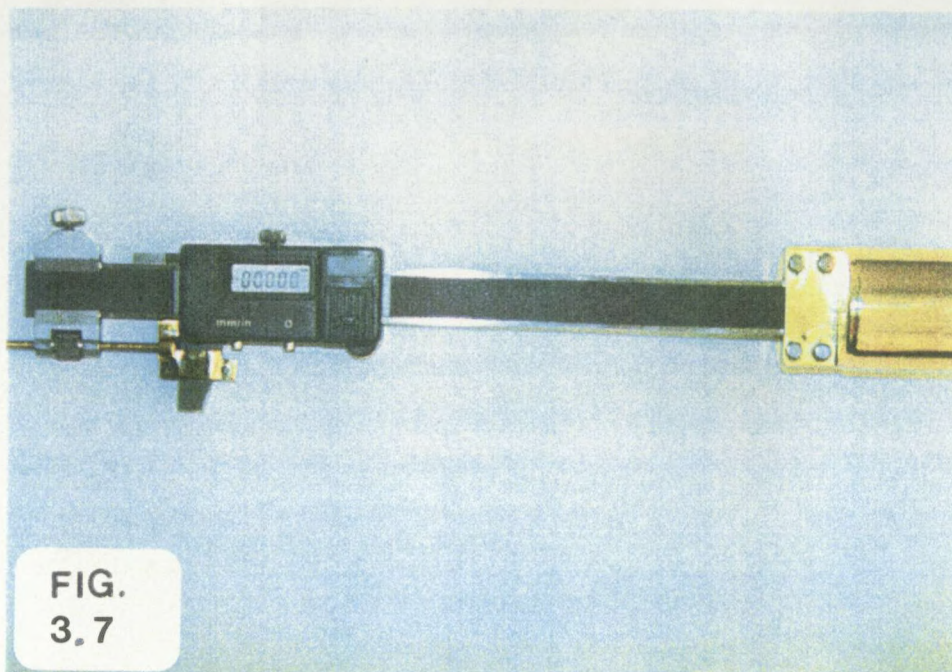
FIGURE 3.2: THE TOP OF THE MEASURING CYLINDER OF THE DEPTH GAUGE

FIGURE 3.3: THE ZEROING OF THE SPECIMEN

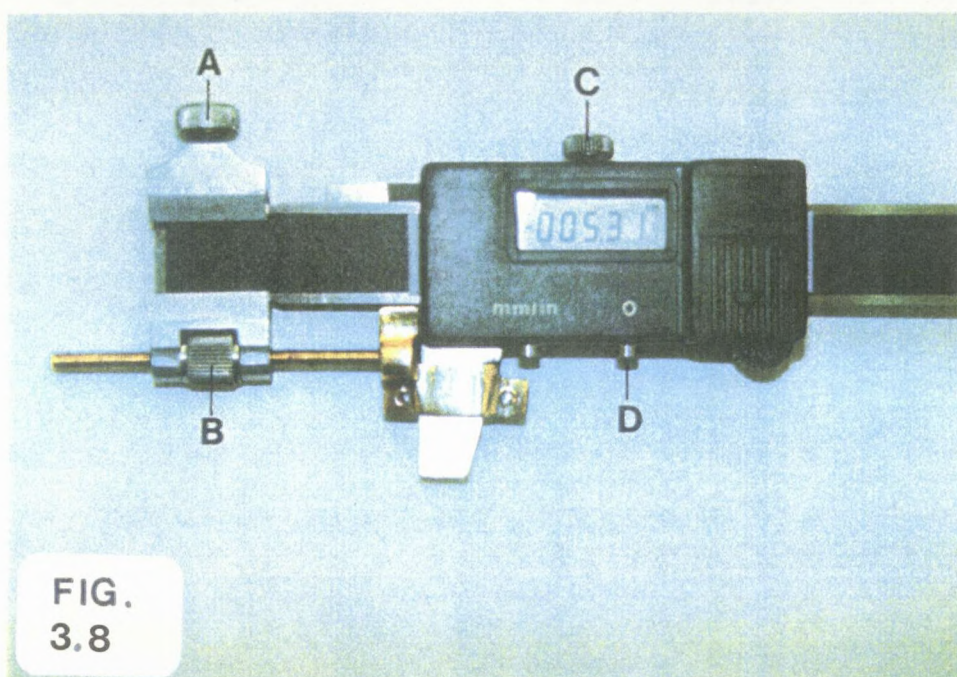
FIGURE 3.4: THE APPLICATION OF CERAMIC TO THE SPECIMEN

FIGURE 3.5: THE CUT BACK OF THE APPLIED CERAMIC

FIGURE 3.6: THE CONDENSING OF THE SPECIMEN USING A CERAMIOSONIC CONDENSER



**FIG.
3.7**



**FIG.
3.8**

FIGURE 3.7: MANUFACTURED DEPTH GAUGE

FIGURE 3.8: DETAILS OF LOCKING MECHANISMS OF DEPTH GAUGE

A - Fine adjustment locking screw

B - Fine adjustment screw

C - Slide locking screw

D - Zeroing button

Before placing the specimen in the measuring cylinder (Fig. 3.2) all the locking screws were released in such a way that the table could move freely inside the measuring cylinder. Following this the specimen was placed on the measuring table, and a sheet of clear plastic perspex was placed over the specimen (Fig. 3.3). The clear plastic sheet was depressed, pushing the specimen down until the plastic sheet was flush with the top of the measuring cylinder. At this stage both the fine adjustment locking screws and slide locking screws were locked and the depth meter was zeroed by pressing the zeroing button on the digital meter (Fig. 3.8). It should be noted that any radical movement or jarring of the depth gauge could cause the reading to change, due to the high sensitivity of the instrument. Therefore, after locking any of the locking screws, the gauge was checked for movement. If any movement was shown then the above zeroing process was repeated.

Once satisfied that the specimen was completely zeroed, the slide locking screw was released and, using the fine adjustment screw, the specimen was lowered to a depth of 1mm. At this stage the Durceram dentine, shade A1 (Ducera, West Germany) ceramic was mixed to a creamy consistency using Vita modeling liquid (Vita, West Germany). The ceramic was then applied to the specimen with a no 6 ceramic modeling brush (Fig. 3.4). Excess moisture in the mixture was removed by blotting the ceramic with a paper blotting towel. The blotting process was then repeated after condensing the dentine on a Ceramosonic Condenser (Shofu Dental Corporation, U.S.A) for ten seconds (Fig. 3.6). To achieve the desired thickness of dentine, a Stanley carving blade was scraped over the top of the measuring cylinder, removing the excess dentine (Fig. 3.5). By turning the fine adjustment screw clockwise, the specimen was removed from the depth gauge and placed on the firing tray. The firing of the dentine layer was in accordance with Table 3.3.

3.4.9.4 MANUFACTURING OF THE CUSTOM SHADE GUIDE

The reflectance spectrophotometer used to test the colour of the ceramic layers was originally designed to test flat specimens. This posed certain questions as to how the colours of the sample groups and the control group were to be related with the shade guides used in the commercial environment, because the shade tabs used are curved and tooth shaped. Therefore a flat custom shade guide was fabricated using the same dimensions as the individual specimens (20mm in diameter).

The preparation of the custom shade guide was done in the same manner as the preparation of the metal-ceramic specimens, with one exception: platinum foil was used as a foundation instead of a non-precious or semi-precious metal. The reason for manufacturing a metal free custom made shade guide was that the commercial shade guide has no metal foundation .

To ensure that the custom made shade guide had the same diameter as the rest of the samples, a piece of platinum foil 35mm x 35mm was wrapped around a spare non-precious specimen, and burnished with a blunt instrument to fit the specimen well. Following this, the application of ceramics was the same as for the rest of the base metal alloy samples. After the firing of the custom made shade guide, the platinum foil was carefully removed with surgical tweezers and then tested in the spectrophotometer using the same procedure as for the base metal alloy samples.

3.4.10 SPECTROPHOTOMETRIC TESTING

3.4.10.1 CALIBRATION

The programme linked to the spectrophotometer is the Datacolour Iris V2.3 (Datacolour AG, Brandbach 10). This programme was specifically designed to test the colours of fabrics used in the textile industry. The selection of options within the programme is discussed in section 3.5.6.1 (C)

Before any testing was done, the spectrophotometer was switched on 15 to 30 minutes before calibration, as recommended by the manufacturers of this instrument. The following settings were used during the calibration and testing of the specimens:

- Diaphragm : 12mm
- Spot : 12
- gloss trap : small
- range : normal

The two calibration standards are a BaSo₄ white standard and a black box standard. The programme for testing the standards was selected and the standards were then tested. The calibration of the spectrophotometer was checked before and after each sample group was tested to ensure that no calibration differences had taken place. For more information about the colourimetric programme, refer to section 3.5.6.1 (C).

3.4.10.2 PROGRAMME SELECTION

After calibration of the spectrophotometer, all specimens were tested using the colourimetry programme. For further explanation as to the access to and uses of this programme, refer to section 3.5.6.1 (b). The following sequence was used to measure the specimens in terms of reflectance, spectral averages and tristimulus (Data Colour Future Manual, 1989):

Task selection : FMP , MEM ,

Sequence of multiple measurement:

- Input code and name (example 0000A) (ceradium)
- Insert specimen
- Start measurement
- Loop back to 2
- End at specimen 30 with F8.

Output options : To print the spectral averages and tristimulus values onto hard copy, the selection was reverted back to the main FMP menu and the following selections were made : SPE, SPM and XYZ followed by PRT (print).

3.4.10.3 TESTING OF THE SPECIMENS

The specimens were removed from their respective boxes in order 1-30, and care was taken that the specimens' surfaces were not touched, especially during the testing of the oxide layers. The reason for this is that the oily deposits from the human finger cause discolouration of the oxide layer. This practice is also recommended by the manufacturers of the respective metal alloys. Following their removal from the boxes, each specimen was individually placed on the testing platform of the spectrophotometer and tested in accordance with the programme instructions. It should be noted that the sample should cover at least 60% of the diaphragm aperture to make the reading valid. Therefore, prior to testing, the specimen was viewed through the viewing aperture to ensure that maximum coverage was achieved. Following this, the specimen was tested, noting at the same time that the xenon lamp exposed twice during testing. If the lamp showed no physical evidence of being fully exposed during testing, then the bulb was checked and the specimen was tested again. Figures 3.9 and 3.10 show one specimen from each sample group ready for testing at the oxide firing, opaque firing and dentine firing stages.

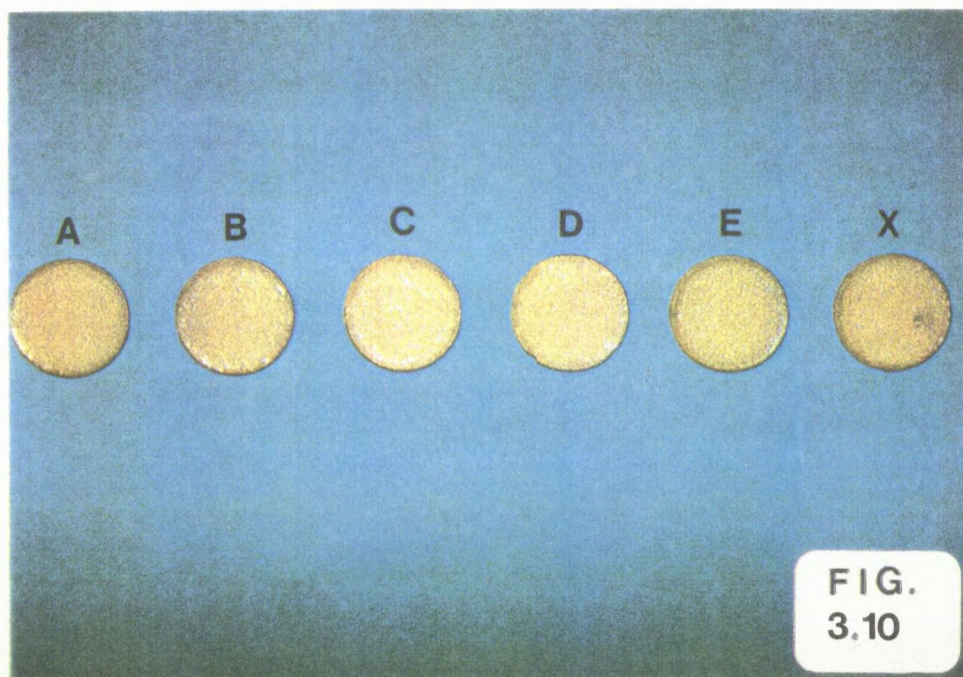
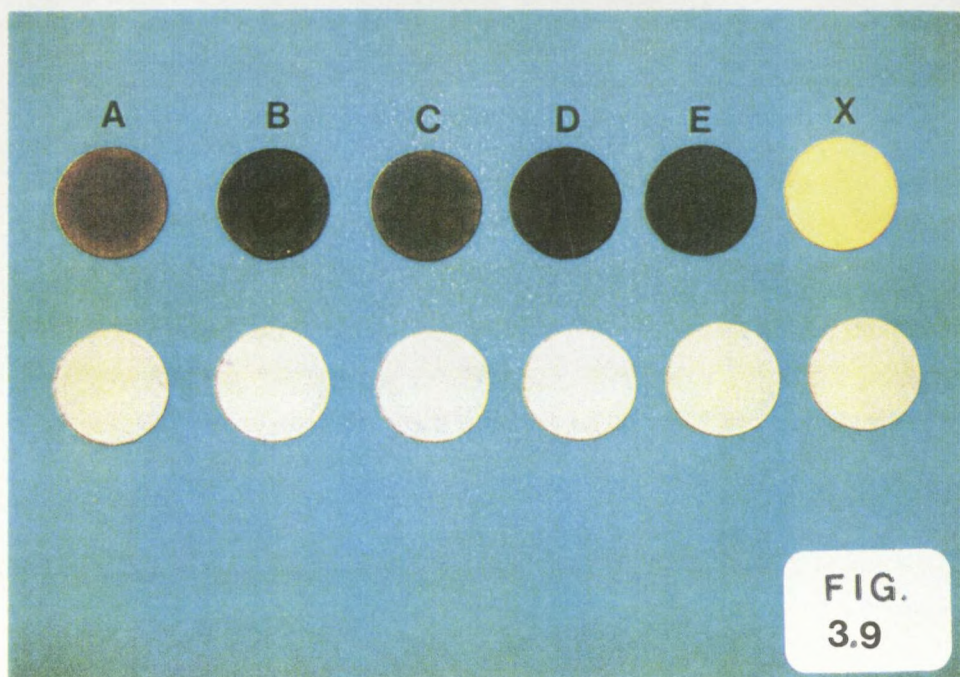


FIGURE 3.9: OXIDE FIRING FOLLOWED BY OPAQUE FIRING

FIGURE 3.10: SPECIMENS AFTER DENTINE FIRING

A = CERADIUM

B = WIRON 99

C = WIROBOND

D = HERANIUM

E = REMANIUM CD

X = BOND-ON-4 (Control Group)

3.4.10.4 PREPARATION OF LIGHT MICROSCOPE SPECIMENS

One specimen was randomly selected from each of the sample groups for microscopic analysis. Following this, each of the selected specimens was carefully sectioned into two, using a diamond separating disc (Renfert Turbo-flex 19 mm, Hilzingen).

To allow for the polishing of the sectioned surface, the specimens were invested in acrylic block (Reditray manufactured by P.G. Smith, Johannesburg), with the surface which was to be polished still visible. A 2mm hole was drilled into the acrylic block on the reverse side which was used as a locator for the polishing arm of the polishing instrument.

Using a Minamet polisher (Buehler LTD, U.S.A), all the specimens were polished using the following polishing times and grit:

<u>Grit</u>	<u>Time(min.)</u>	<u>Speed level</u>
400	6	5
600	6	5
1000	8	6
Diamond paste	8	7

Note: Speed level = The number stipulated on the Minamet polisher (Buehler LTD, U.S.A).
Using a Leica Leitz DM RM (Germany) light microscope, the specimens were photographed at a magnification of 200x life size and analysed (Annexure A).

3.4.10.5 STATISTICAL ANALYSIS

Due to the fact that colour is interpreted in a three dimensional form, the statistical formulations were complex. The data output produced by modern spectrophotometers is normally processed statistically by the software integrated into the spectrophotometer system.

The mean reflectance values (SPE) and CIE LAB (XYZ) tristimulus values were calculated by the spectrophotometer computer and were printed out in hard copy (Tables 4.1-8).

The mean spectral reflectances were then subjected to the two-sample analysis test (Annexure B). The five base metal alloys were then statistically compared with one another and with the gilded control group. From these calculations, the P-values, the computed t-statistic and the variances were obtained.

In order for individual recognition of the sample groups during the statistical testing stages, each sample group was given a statistical number.

For example: Ceradium V (S1) = oxide firing
(S21) = opaque firing

3.5 DISCUSSION OF IMPORTANT FACTORS THAT AFFECTED THE MATERIALS AND METHODS

3.5.1 INTRODUCTION

The following section is a discussion as to why certain techniques, materials or items of machinery were used. The importance of this section is that it offers an explanation of methods used which may not be conventional. A second reason for this section is that there are possible aspects of this study which may have been otherwise affected, if different techniques had been used. This section should be read in conjunction with section 3.4, which deals with the methodology.

3.5.2 FINGER PUNCH

In order to produce specimens that were of the exact same dimensions, a specially engineered finger punch was produced (Fig. 3.1). The punch was manufactured out of brass to avoid any corrosion of the instrument during the study.

The punch was fabricated in two pieces (Fig. 3.1):

- the cylindrical cutting tube (A2)
- the pattern plunger (A1).

The inside diameter of the cylindrical cutting tube was machined with a light taper from 21mm to 20mm towards the cutting edge. The reason for the taper was to ensure that the pattern plunger did not slide all the way through the cylindrical cutting tube, which might have damaged the specimen.

The pattern plunger was fabricated to allow the removal of the wax specimen without distorting it. It was found that the wax specimen of 0.4mm was too thin to remove successfully from the cylindrical cutting tube by hand.

The punch was used in a simple manner similar to that of a leather punch, from which this idea originated. The pattern plunger was removed before cutting to ensure that the cutting of the pattern could be monitored from inside the cylindrical cutting tube. The cutting of the pattern was achieved by placing an 0.4mm wax sheet on a flat surface and depressing the cutting cylinder by hand until the pattern was cut. At this point the plunger was depressed into the cutting cylinder to carefully remove the pattern.

3.5.3 THE LAYERING DEPTH GAUGE

In the literature related to this study, there was very little reference to instrumentation used to acquire specific depth of ceramic. Although there has been extensive research using ceramic materials in certain studies, most of which having included bond strength of the metal ceramic restoration, researchers (Goodkind, *et al.*, 1985; Judson, *et al.*, 1985) have consistently failed to determine how exact depth of ceramics could be achieved.

When dealing with colours in ceramics, one of the greatest dependents is the amount of ceramic applied to the metal foundation. Further-more, most of the resultant colours in metal ceramics come from refraction, reflection and dispersion of the incident light. Therefore, by the laws of physics, most of the latter are dependent on the depth of the medium which the light is transmitted through.

Because porcelain is a difficult medium to work with in terms of firing shrinkage (volumetric and linear shrinkage), it would be highly assuming if one were to place arbitrary amounts of porcelain on the metal alloy surface. Therefore it was decided that the porcelain would be applied using a depth gauge that could accurately measure up to two decimal places (Fig. 3.7). The instrument

chosen was a modified digital vernier (Mayoto, 150mm digital vernier, Japan). The vernier had the following specifications:

- Resolution : 0.01mm
- Accuracy : $\pm 0.03\text{mm}$
- Measuring system : non-contact linear CAP
- Data output: serial output for interface with host computer printer
- Display : LCD.

The manufactured portion of the depth gauge proved to be the most critical portion of the instrument (Fig. 3.3). The depth gauge cylinder and piston were manufactured out of brass to reduce the possibility of corrosion, especially during ceramic application which has a fair amount of moisture content. The inside diameter of the cylinder was machined out to 20mm to be able to accommodate the specimens. The piston was then made to fit neatly inside the cylinder.

The locking mechanism (Fig. 3.8) was made to enable the piston to be locked into position during ceramic application and to produce repeatable results in terms of measurement. To allow fine adjustment of the depth gauge, a fine adjustment screw was added to reduce the time taken to reach the exact measurement required on each specimen.

3.5.4 INDUCTION CASTING SPECIFICATIONS

Due to the complexity of the induction casting process, it is important to mention the specific settings of the induction casting machine (Galloni 3N Modular, Italy) used in the experimentation.

Following the manufacturer's recommendation, the casting machine was turned on at least five minutes before the actual casting process. This allowed for the warming of the electrical components of the machine which in turn ensured repeatable results.

For this experimental process the following settings were used:

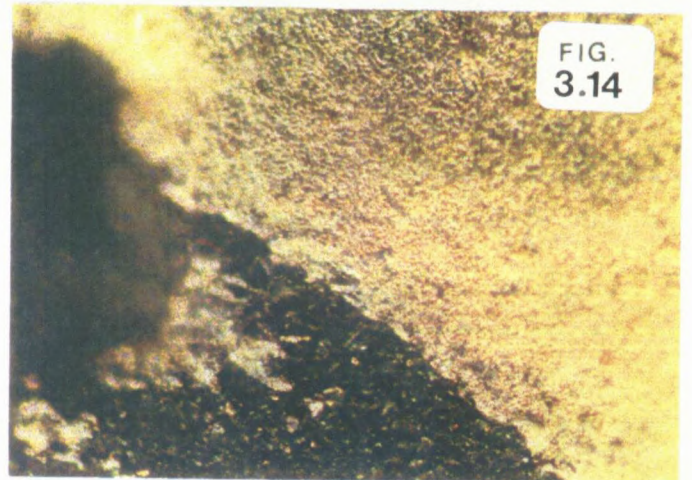
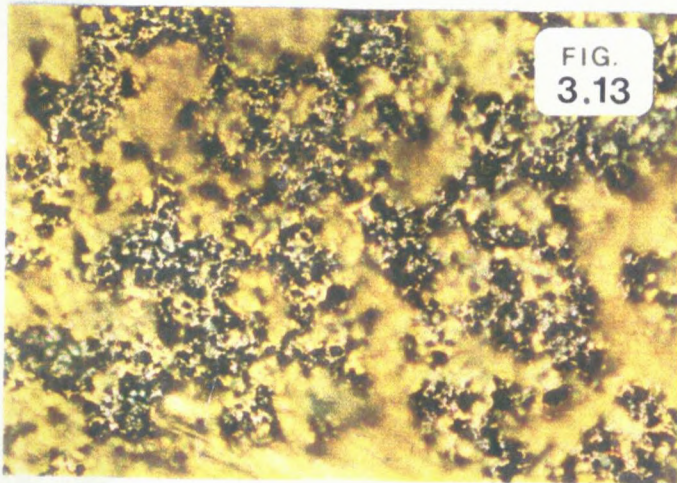
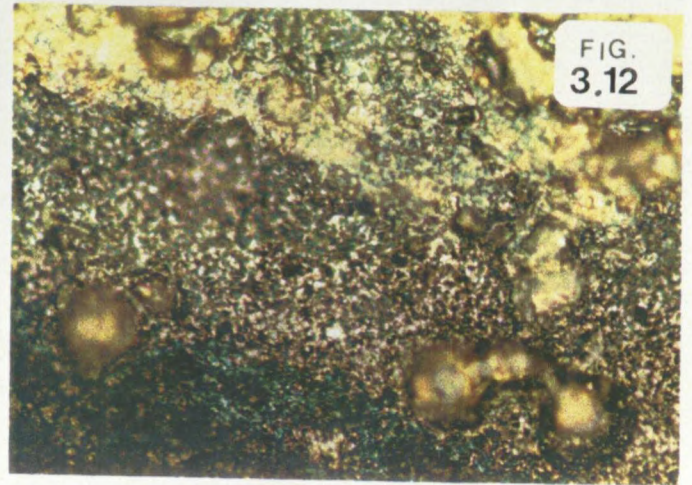
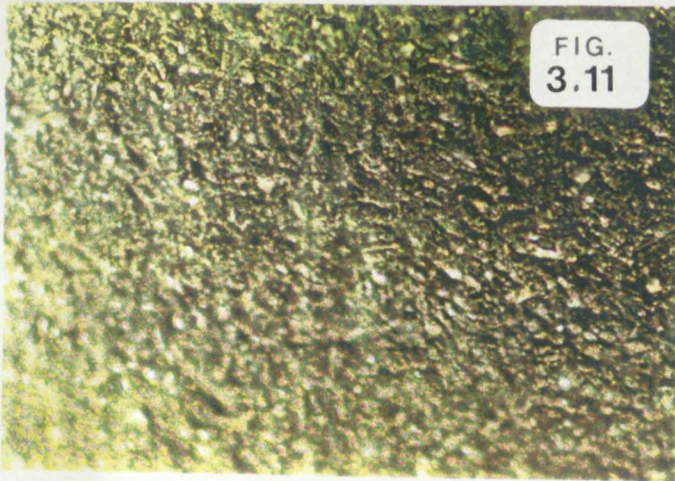
- The crucible setting was set on No 2 for ceramic crucibles only.
- Induction power was set to 1500Hz.
- The stabilizing switch was set to the **on** position before the heating of the alloy.
- Only when audible clicking was heard, was the selector switch changed to the **free** position.

3.5.5 APPLICATION OF CERAMICS

3.5.5.1 SPRAYING OF THE OPAQUE LAYER

When faced with the problem of which form of opaque application to use, there were two possible options: the first being the direct application of opaque by means of a brush, and the second being the spraying of opaque with an air brush. Lourenstine (1993) commented that the spraying of opaques had many advantages, of which the most important were that the metal coloured oxides were blocked out more effectively, and that the technique was very time efficient. The latter was a very important factor aside from the colour effects due to the fact that the sample groups were very large.

Consistency in opaque however was the prime objective and it was argued that, due to the difficulty in controlling this factor by hand, spraying of the opaque onto the specimen was a better option. The layering of the opaque material could also be judged more accurately with the spray application method.



TESTING OF THE GILDING MATERIALS

FIGURE 3.11: THE OXIDE LAYER OF A BASE METAL ALLOY

FIGURE 3.12: THE RESULT OF DECK GOLD FIRING

FIGURE 3.13: THE RESULT OF SPECTRA-SEAL FIRING

FIGURE 3.14: HALF OXIDE, HALF SPECTRA-PASTE FIRING

The alternate method of applying the opaque material would be by hand with a ceramic brush. The problem with applying ceramic using this method is the fact that the pressure of the brush stroke leaves striations on the finished layer. These marks are areas that have not been fully covered with material and the result is dark lines being left on the surface after firing. It was thought that it could affect the resultant colour of the finished restoration. It is interesting to note that during the inquiry as to which method of opaque application would be more suitable, it was found that at least 75% of the commercial environment applied opaque by hand using the brush-on technique. This would be an interesting line to study in future research projects.

3.5.5.2 TESTING OF THE GOLD GILDING MATERIALS FOR THE CONTROL GROUP

During the cost budgeting of this study, it was assumed that most of the gilding materials provided in the commercial industry today were of the same quality and reliability. However, during the process of selecting a gilding application material, it was found that the results did not comply with the requirements of this study. It was for this reason that a pilot study was undertaken to find a suitable gilding material that would produce adequate results.

The following were the requirements for the material to be accepted as suitable for the gilding of the control group:

- The gilding material should produce a gold coloured matt finish covering the specimen surface entirely.
- The material should remain inert after firing and only be involved in enhancing the bonding.
- The oxide of the metal alloy should not affect the gilding material colour in any way and the gilding material should mask out the affects of the dark oxide layer (Fig. 3.11).
- The application and firing of the gilding material should not be technique sensitive.
- The material should be cost effective.

Three locally available brands of gilding materials were tested and evaluated in terms of above requirements. The three brands were as follows:

- Brand A: VITA SPECTRA-GOLD Manufactured by Vita Zahnfabrik, Germany.

This product, which is available in a 4 g syringe, is pre-mixed to a paste form. The recommended firing cycle is as follows:

- pre-drying temp. : 500-550° C
- holding time : 0min.
- climb time : 3min.
- top temp holding time : 1min.
- top temp. : 820° C
- vacuum : 0%

- Brand B: SPECTRA-SEAL manufactured by Unitek, Monrovia, CA.

Spectra-seal is supplied in powder and liquid activator form and is mixed to a creamy consistency and applied directly to the metal surface. The firing programme is as follows:

- pre-drying temp. : 500°C - 550°C
- holding time : 5min.
- climb time : 55°C/ min.
- top temp. holding time : 0min.
- top temp. : 700°C-1010°C
- vacuum : 100%

- Brand C: DECK-GOLD (normal) manufactured by Degussa, Germany.

Deck-gold is supplied in already mixed form in an easy to use container with a supplied brush. The firing times and temperatures are the same as for Spectra-Seal.

Although the manufacturers' have produced much information on their products and their uses, a pilot study was done exactly in accordance to the manufacturers recommendations. However, the following report is not an in-depth study of the materials and their flaws. The purpose of this investigation was to find a suitable material which could fulfil the requirements of the control group in the original study. The reason for presenting this section is to enlighten the reader as to the wide variety of gilding materials available today, and possibly guide other researchers as to which materials may produce undesirable results in their endeavours. The results were as follows:

Only one out of three brands produced a satisfactory layer which complied with the requirements previously mentioned (section 3.5.5.2). The brand which produced this desirable result was brand A (Fig. 3.14). In terms of colour, brand A produced a matt gold foundation which was conducive to strong mechanical bonding and a warm gold coloured finish, as opposed to the finishes of brand B and C which produced dark finishes with indiscriminate areas of material (Fig. 3.12 and 3.13).

The results of the light microscope investigation showed that brands B and C suffered from lack of structural cohesion. It was found that brand B produced a gold lattice (Fig. 3.13) which lacked wetting abilities on the surface of the metal. It was thought at this stage that a possible reason for this phenomenon was that the firing temperature was not sufficiently high to melt the material into a cohesive mass. An attempt was made to burnish the lactic of brand B to see if the result would produce a golden matt finish. Although the attempt at burnishing was successful, the colour remained dark and there was a certain amount of concern with regard to the damage that burnishing had on the bond strength of the metal ceramic interface. However, after raising the temperature to the maximum recommended temperature, there was no significant change in structure. Brand C also produced wetting problems; but more important, was the colour of the gilding material (Fig. 3.12). Upon closer magnification it was found that there was substantial saturation of the material into the oxide layer and contamination of the gilding layer. Figure 3.12 shows the border between the oxide and the gilding material of brand C. Note the blue tinting of the gold. The manufacturer recommends this brand for the sealing ability of the metal substructure, which is possibly the reason for the deep penetration of the gilding material into the oxide layer.

The manufacturers of brands B and C recommend that if deeper gold colour and enhanced bond strength are required, then it is possible to apply the gilding layer after the first opaque layering. The latter was experimented with and the results produced the same flaws as when the normal layering process was used.

Upon searching for a possible link in the reason for the incoherent formation of brands B and C, light microscope tests were done on the raw ingredients of the two brands. It appeared that the two brand types had almost the same basic gold particle size and shape, which lead us to the possible fineness of the gold and the fluxing solutions. However, these

were beyond the scope this study and could possibly lead to an interesting study in the future.

In summary, brand A produced a cohesive mass of a warm golden colour which was satisfactory for the gilding process. Brands B and C produced inconsistent results even though the furnace temperatures were adjusted in an attempt to find solutions to the anti-fluxing phenomenon of the gold. All brands were provided in a kit form and were easy to use.

3.5.5.3 THE SELECTION OF DUCERAM CERAMIC

Although popularity, cost and availability were significant factors in the selection of Duceram (the porcelain powders used in this study), one of the more important factors influencing the final decision was the unique advantages that Duceram has to offer.

Firstly the Duceram powders are produced in a manner that has the pigment melted into the powder frit, which is then subsequently ground into powder. This process ensures that the porcelain will not devitrify and lose its colour after multiple firings. Secondly, due to the nature of this process, the pigment does not 'settle' out of the powder like more conventional porcelains after standing or transportation.

3.5.6 SPECTROPHOTOMETER CONSIDERATIONS

3.5.6.1 DESCRIPTION OF SPECTROPHOTOMETER

(a) PHYSICAL STRUCTURE OF THE SPECTROPHOTOMETER

The TEXFLASH (DATA COLOUR- COLOUR FUTURE, Dietlekon AG) reflectance spectrophotometer is a compact dual-beam spectrophotometer with a measurement geometry specially designed for the textile industry. Exchangeable measurement diaphragms, gloss mask and continuously variable optics enable the user to adapt the measurement optimally to the specimen being measured.

The specimen is diffusely illuminated by a pulsed xenon lamp, and viewed at an angle of 0°.

A continuously variable proportion of the radiation below the visible range can be filtered out by means of the integrated UV calibration system.

A spring loaded support presses the specimen up against the measuring aperture and the specimen can be checked for alignment through a viewing aperture.

Every time the specimen is measured it is compared by 16 measurement values with reference areas in the measurement area in the range of the visible radiation (400nm - 700nm). Microprocessors control the measurement sequence, compute the results and transfer the data to a standardized interface.

(b) THE SPECTROPHOTOMETER PROGRAMME SELECTION

Within the data colour programme there are many textile programmes, some of which include dyeing corrections and various dyeing programmes. These will obviously not be included in this section. Most of the testing done in this study involved the colourimetric programme, and the selections used in this study follow. It should be noted that all the selections in the programme are abbreviated when appearing on the screen; these abbreviations are used in the text of this study, the reason being purely for the researcher's convenience.

The colourimetry selections are as follows:

(c) COLOURIMETRY PROGRAMME OPTIONS

FMP: colourimetry programme

KAL: calibration

MEM: multiple measurement

SPE: spectral value

SPM: average spectral values

XYZ: tristimulus values

LST: list

PRT: print

CHAPTER FOUR

RESULTS

4.1 REFLECTANCE RESULTS

4.1.1 INTRODUCTION

Before attempting to read the results in the section to follow, it is important that the reader understands objective colourmetric analysis. There is a difference between subjective and objective colourmetric analysis. Subjective colour evaluation is the perception of colour by the human eye and is biased when comparisons of similar colours are perceived. This has been noted widely throughout the literature. Objective colour testing utilizes scientific instrumentation and avoids the human element of bias.

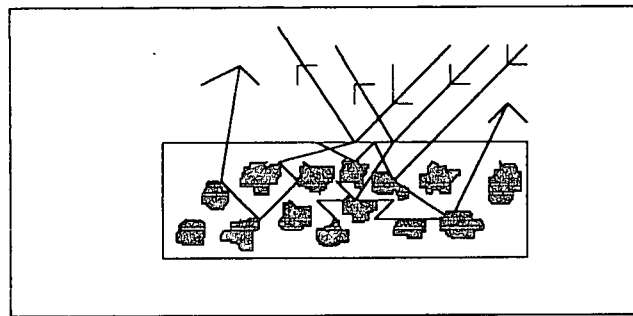


FIGURE 4.1: MICROSCOPIC REFLECTION AND SCATTERING OF LIGHT

Lund *et. al.* (1985: 794) notes that the actual perception of colour is based on whether the reflected light is affected by subtractive mixing or additive mixing (Fig. 4.1).

- Subtractive mixing takes place when certain wavelengths of light are selectively absorbed one after another and the result produces a certain colour.

- Additive mixing on the other hand produces a resultant colour due to the scattering of small different colour pigments, and because these colour differences are so small the object is perceived to have only one colour. Lund and associates noted that the colour of ceramic material is a combination of both additive and subtractive colour mixing (Fig. 4.1).

Yamamoto (1985: 221) reminds us that light in the visible range is a combination of wavelengths from 400nm to 700nm in length. The spectrophotometer used in this study, objectively measured the colour of the specimens and produced 32 measurements of reflected light between the 300nm and the 700nm wavelengths. The measurement in the reflectance tables (Tables 4.1, 4.3, 4.5 & 4.7) is expressed as percentages of reflected light. As stated previously, reflected light is affected by additive, subtractive and absorption of light. In other words, this is the composition of the shade in terms of the wavelengths of light reflected. The figures in the reflectance tables (4.1, 4.3, 4.5 & 4.7) increase uniformly from left to right, in the shape of a graphical curve. The shapes of the curves indicate which range of colours is reflected and which is absorbed, and this is graphically illustrated (Fig. 4.2, 4.3 & 4.4). These reflectance curves allow the reader to understand the **composition** of the reflected colour as opposed to the tristimulus and chromaticity values (Tables 4.2, 4.4, 4.6 & 4.8) which locate the **colours' position** on a three dimensional colour form (DIN 5033 (1964), Annexure C). This three dimensional colour form is illustrated graphically as the Hue, the Chroma (Fig. 4.5, 4.7, 4.9 & 4.11) and the Value (the relative lightness or darkness of a colour, or the quality of grey) (Fig. 4.6, 4.8, 4.10, & 4.12).

To allow for better comprehension of the spectrophotometer results, section 4.1 deals with the results in terms of reflectance versus wavelength, and section 4.2 reports the specimens' results in terms of their chromaticity values. The reason for separating the two sections is that the reader will have a better understanding of the specimens' change in colour, not only in terms of wavelength, but also with regard to the positioning of the specimen on the chromaticity diagram (DIN 5033, 1964, S.A.B.S) (Annexure C).

It must be noted, that objective colourimetric analysis was used in this study, and all possible tools were used to produce valid results. The reader must be reminded that people rather than machines must get involved in the final steps of the colour assessment procedure (Billmeyer, 1981:69).

Table 4.1: Mean Reflectance Values of Base Metal Oxide Firing

METAL ALLOY (*)	WAVE- LENGTH (nm)	REFLECTANCE %									
		0%	10%	20%	30%	40%	50%	60%	70%	80%	90%
Ceradium (S2) P=0 (S3) P=0 (S4) P=0 (S5) P=0	300										11.30
	400	11.56	11.82	12.07	12.30	12.54	12.79	13.04	13.28	13.52	13.76
	500	13.99	14.20	14.38	14.54	14.69	14.82	14.95	15.07	15.20	15.36
	600	15.52	15.65	15.98	15.98	16.19	16.40	16.64	16.90	17.17	17.42
	700	17.66									
Heranium (S3) (S4) (S5)	300										7.18
	400	7.33	7.48	7.52	7.38	7.25	7.25	7.26	7.38	7.61	7.88
	500	8.18	8.48	8.76	8.98	9.13	9.20	9.21	9.18	9.15	9.21
	600	9.30	9.43	9.58	9.78	10.00	10.24	10.47	10.68	10.88	11.06
	700	11.24									
Remanium CD (S3) (S4) (S5)	300										9.80
	400	9.76	9.73	9.63	9.24	8.85	8.72	8.73	8.91	9.21	9.58
	500	9.98	10.40	10.71	10.83	10.81	10.57	10.23	9.86	9.54	9.39
	600	9.33	9.34	9.41	9.56	9.74	9.88	10.06	10.40	10.77	11.08
	700	11.37									
Wirobond (S5)	300										11.24
	400	11.21	11.17	11.07	10.60	10.15	10.02	10.04	10.30	10.71	11.23
	500	11.80	12.38	12.86	13.11	13.17	12.96	12.63	12.02	11.93	11.82
	600	11.81	11.88	12.04	12.27	12.54	12.78	13.06	13.04	13.79	14.11
	700	14.40									
Wiron 99	300										7.89
	400	8.17	8.54	8.59	8.54	8.47	8.50	8.61	8.84	9.14	9.50
	500	9.88	10.23	10.51	10.69	10.77	10.47	10.66	10.53	10.44	10.44
	600	10.44	10.49	10.58	10.70	10.84	10.99	11.15	11.26	11.37	11.50
	700	11.64									

(*) Indicates the statistical number allocated to each metal alloy , followed by their P values.

Table 4.2: Tristimulus Values of Oxide Firing

METAL	ILLUMINANT	X	Y	Z	x	y	Y
Ceradium V	D65	14.12	14.78	13.79	.3307	.3462	14.7768
	A	17.13	15.08	4.58	.4656	.4100	15.0818
Heranium	D65	8.41	8.81	7.82	.3358	.3520	8.8142
	A	10.27	9.04	2.59	.4691	.4128	9.0403
Remanium CD	D65	9.14	10.01	9.74	.3162	.3466	10.0119
	A	10.75	9.94	3.21	.4497	.4160	9.9434
Wirobond	D65	11.22	12.16	10.97	.3265	.3540	12.1603
	A	13.46	12.24	3.63	.4889	.4174	12.2437
Wiron 99	D65	9.69	10.38	9.36	.3292	.3527	10.3782
	A	11.69	10.51	3.11	.4619	.4153	10.5097

OXIDE FIREING

SHOWING PERCENTAGE REFLECTANCE

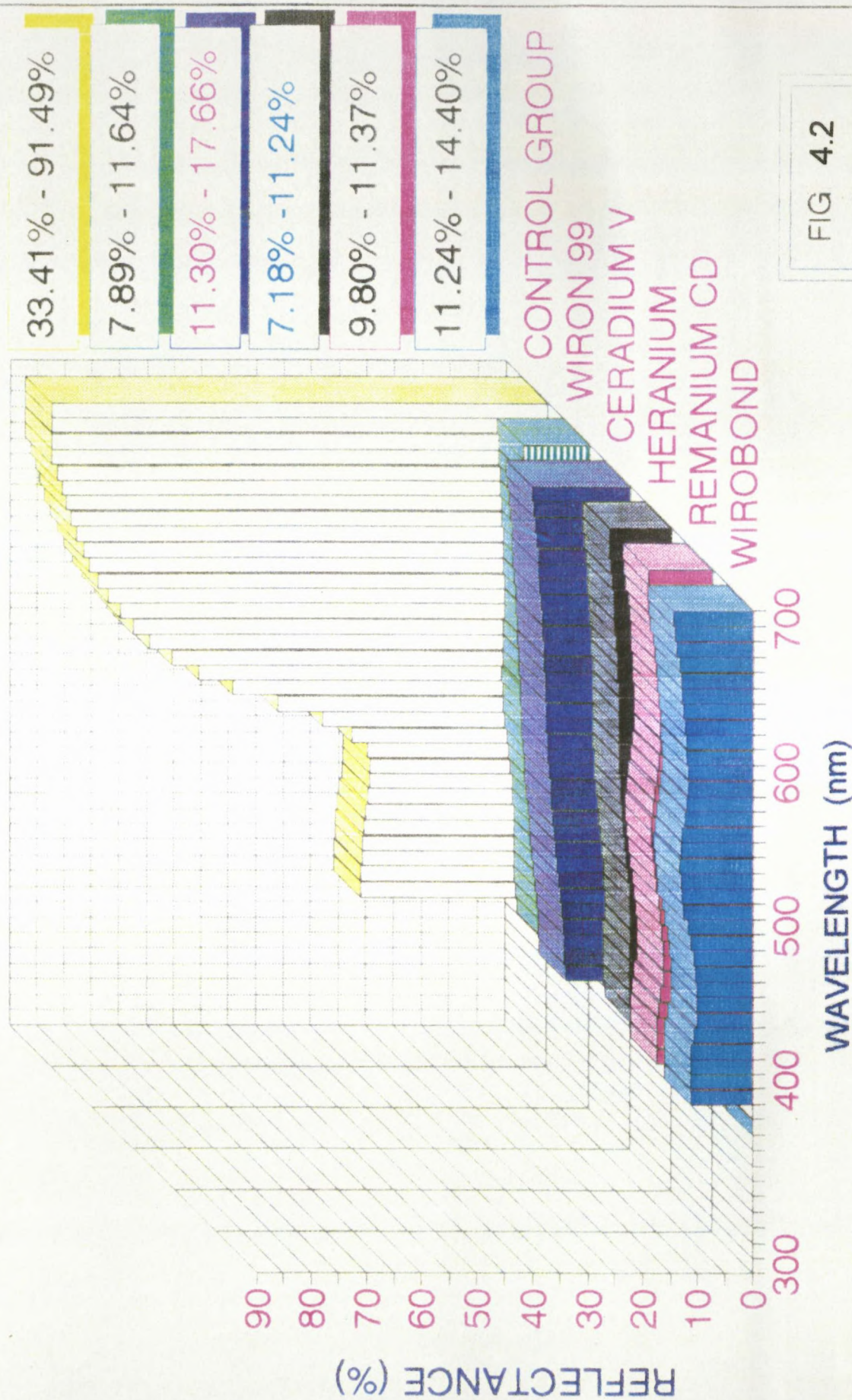


FIG 4.2

4.2 SUBPROBLEM ONE

4.2.1 OXIDE REFLECTANCE CURVES

Since the oxide layer is the foundation that provides the chemical and physical bonding of the metal alloy and the ceramic, virtually all manufacturers advocate the firing of an oxide layer. Therefore the colour of the oxide layer is of crucial importance to the ceramist with a trained eye for colour. In this study it was hypothesized that there were definite differences in the colours of the base metal alloy oxide layers.

Table 4.1 lists the five base metal alloys used in this study. For each base metal alloy, 32 reflectance readings were produced by the reflectance spectrophotometer. The 32 reflectance measurements were obtained between the 300nm and the 700nm visible range. To obtain the percentage of light which was absorbed by the specimen, the reflected percentage was subtracted from 100%, for example:

$$100\% (\text{Visible light}) - 11.30\% (\text{Reflected light}) = 88.70\% (\text{Absorbed}).$$

It should be noted that the reflectance values were more important to the results than the absorbed values, since the reflectance values reported the actual visible colour.

Figure 4.2 is a graphic representation of Table 4.1. The graphs in figure 4.1 are visual conception media to define the nature and composition of the oxide colours. In terms of reflectance colour of the oxide layer (Table 4.1 and Fig. 4.2), all the reflectance curves produced different wavelength patterns. The curves in Figure 4.1 show percentage reflectance versus wavelength. The shape of each series gradually increases in nature with various fluctuations between 300nm and 700nm, indicating that there were different wavelengths of colour within one specific metal oxide.

Wiron 99 produced a reflectance percentage of between 7.89% (300nm) - 11.64 % (700nm) and a deviation of between 0,56% - 0,93%. However, Wiron 99 produced no apparent fluctuations in terms of wavelength between 300nm - 700nm. Wiron 99 reflected results which suggested reflectance values which were darker than those of Ceradium V which in turn produced a reflectance curve which was also monochromatic in colour with a reflectance value of between 11.30% (300nm) - 17.66% (700nm) with a deviation range of between 0.56% - 0.93%. Heranium had a reflectance value of between 7.18% - 11.24%, which was similar to that of Rermanium CD which had a reflectance value of 9.80% - 11.37%. Although there was a slight

difference in reflectance values the similarity could be confirmed visually. Wirobond was lighter in colour than Rermanium CD, Heranium and Wiron 99; but with a reflectance value of between 11.24% - 14.40%, it was darker than Ceradium V. Although Wirobond and Rermanium CD produced comparable high and low reflectance values, their reflectance curves both showed fluctuation around the 530nm wavelength. The increase of reflectance values in the 530nm wavelength suggests that there was an increase in the green reflectance wavelength.

A discussion on the gilded semi-precious control group has not been included in the section on the oxide results because of the obvious colour differences between the gold coloured control group and the dark coloured oxide layer of the base metal alloys.

Table 4.3: Mean Reflectance Values of Opaque Firing

METAL ALLOY (*)	WAVE- LENGTH (nm)	REFLECTANCE %									
		0%	10%	20%	30%	40%	50%	60%	70%	80%	90%
Ceradium (S21) P=0.52338 ACCEPTED	300										41.94
	400	42.10	42.27	42.70	43.74	44.38	45.65	47.14	48.76	50.52	52.44
	500	54.37	65.21	57.86	59.15	60.22	61.20	62.07	62.81	63.50	64.25
	600	64.97	65.65	66.18	66.44	66.47	66.22	65.87	65.60	65.63	65.15
	700	64.97									
Heraium (S22) P=0.459989 ACCEPTED	300										42.43
	400	42.61	42.78	43.21	43.90	44.86	46.13	47.62	49.26	51.04	52.97
	500	54.92	57.77	58.43	59.70	60.76	61.73	62.59	63.31	63.99	64.72
	600	65.42	66.09	66.61	66.86	66.89	66.65	66.32	66.06	65.04	65.64
	700	65.47									
Rermanium CD (S23) P=0.331704 ACCEPTED	300										41.82
	400	41.99	42.16	42.59	43.29	44.25	45.51	46.99	48.61	50.36	52.25
	500	54.16	55.97	57.58	58.81	59.83	60.77	61.58	62.27	62.90	63.59
	600	64.25	64.87	64.35	65.58	65.59	65.34	65.00	64.75	64.53	64.35
	700	64.20									
Wirobond (S24) P=0.148489 ACCEPTED	300										41.39
	400	41.60	41.82	42.27	42.94	43.85	45.07	46.48	48.02	49.68	51.45
	500	53.22	54.90	56.39	57.49	58.39	59.22	59.93	60.51	61.04	61.61
	600	62.16	62.69	63.10	63.28	63.28	63.05	63.24	63.45	63.61	64.53
	700	64.97									
Wiron 99 (S25) P=0.520962 ACCEPTED	300										41.94
	400	42.10	42.27	42.70	43.74	44.38	45.65	47.14	48.76	50.52	52.44
	500	54.37	65.21	57.86	59.15	60.22	61.20	62.07	62.81	63.50	64.25
	600	64.97	65.65	66.18	66.44	66.47	66.22	65.87	65.60	65.35	65.15
	700	64.97									
Bond-on-4 (Control group)	300										42.11
	400	42.28	42.44	42.88	43.58	44.57	45.87	47.40	49.09	50.94	52.99
	500	55.08	57.11	58.97	60.44	61.71	62.90	63.99	64.96	64.91	66.96
	600	68.00	69.00	69.82	70.29	70.47	70.26	69.91	69.68	69.47	69.31
	700	69.19									

(*) Indicates the statistical number allocated to each metal alloy, followed by their P values.

Table 4.4: Tristimulus Values of Opaque Firing

METAL	ILLUMINANT	X	Y	Z	x	y	Y
Ceradium V	D65	56.97	59.95	50.85	.3396	.3573	59.9541
	A	69.93	61.61	16.99	.4708	.4148	61.6129
Heranium	D65	57.74	60.64	50.48	.3419	.3591	60.6389
	A	71.14	62.49	16.88	.4726	.4152	62.4923
Remanium CD	D65	56.76	59.67	49.79	.3415	.3590	59.6674
	A	69.88	61.45	16.65	.4722	.4152	61.4480
Wirobond	D65	55.19	58.14	49.25	.3395	.3576	58.1426
	A	67.73	59.73	16.46	.4706	.4150	59.7278
Wiron 99	D65	57.29	60.13	49.94	.3423	.3593	60.1287
	A	70.62	62.00	16.71	.4729	.4152	61.9959
Bond-on-4 (Control group)	D65	59.43	61.97	50.25	.3462	.3610	61.9735
	A	73.78	64.23	16.82	.4764	.4149	64.2551

4.3 SUBPROBLEM TWO

4.3.1 OPAQUE REFLECTANCE CURVES

The opaque layer was applied to conceal the effects of the oxide layer, or in the case of the gilded control group, the effects of the gilded layer. Therefore the gilded semi-precious control group was introduced into the testing process only at this stage.

Unlike the reflectance curves of the oxide firings, the opaque reflectance curves (Table 4.3 and Fig. 4.3) were similar in nature and differences in the reflectance values were insignificant. This was confirmed statistically by a 95% confidence level with P - values (Table 4.3), with the null hypothesis being accepted for all. Visually, however, there were differences, which are explained in chapter five.

The gilded control group produced a reflectance value of between 42.11% - 69.19%, with a deviation range of between 0.36% - 0.75%. The control group displayed the highest reflectance of all the opaque readings, specifically in the 650nm - 700nm wavelength. This increase indicates that there was a stronger presence of longer wavelengths of (red) light than with any of the non-precious metals.

OPAQUE FIRING

SHOWING PERCENTAGE REFLECTANCE

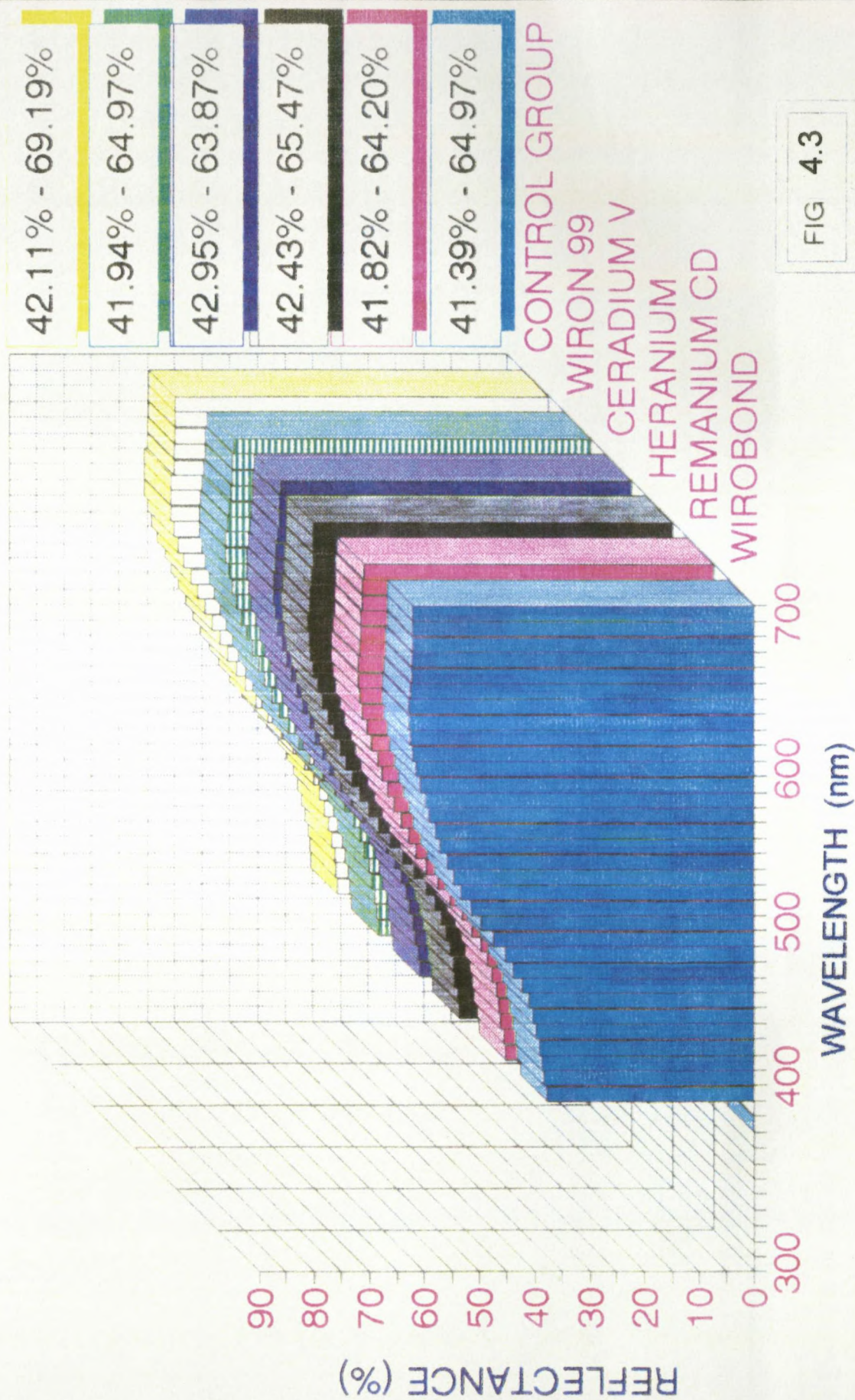


FIG 4.3

Table 4.5: Mean Reflectance of Dentine Firing

METAL ALLOY	WAVE- LENGT H (nm)	REFLECTANCE %									
		0%	10%	20%	30%	40%	50%	60%	70%	80%	90%
Ceradium (S31) P=0.552538 ACCEPTED	300										38.69
	400	39.08	39.46	39.94	40.50	41.18	42.00	43.08	44.6	46.49	48.70
	500	50.93	52.86	54.57	56.05	57.34	58.48	59.53	60.5	61.52	62.48
	600	63.35	64.05	64.56	64.83	64.88	64.64	64.32	64.0	63.90	63.76
	700	63.65									
Heranium (S32) P=0.686055 ACCEPTED	300										38.75
	400	39.13	39.51	39.98	40.52	41.19	42.02	43.12	44.6	46.56	48.79
	500	51.07	53.08	54.89	56.44	57.80	59.01	60.11	61.1	62.21	63.22
	600	64.14	64.90	65.46	65.76	65.84	65.62	65.30	65.1	64.95	64.83
	700	64.74									
Remanium CD (S33) P=0.271595 ACCEPTED	300										38.72
	400	39.09	39.46	39.92	40.43	41.06	41.82	42.85	44.3	46.15	48.31
	500	50.51	52.42	54.14	55.63	56.94	58.10	59.16	60.1	61.16	62.12
	600	62.98	63.69	64.21	64.49	64.54	64.32	64.02	63.8	63.70	63.60
	700	63.54									
Wirobond (S34) P=0.283881 ACCEPTED	300										38.37
	400	38.75	39.13	39.58	40.10	40.74	41.51	42.56	44.0	45.88	48.03
	500	50.21	52.08	53.75	55.17	56.42	57.51	49.00	59.4	60.32	61.19
	600	61.98	62.62	63.10	63.34	63.38	63.16	62.86	62.6	62.55	62.45
	700	62.38									
Wiron 99 (S35) P=0.593272 ACCEPTED	300										38.46
	400	38.82	39.19	39.65	40.18	40.84	41.65	42.73	44.2	46.18	48.42
	500	50.71	52.73	54.54	56.12	57.50	58.74	59.87	60.9	62.03	63.09
	600	60.04	64.83	65.41	65.73	65.80	65.57	65.24	65.01	64.83	64.6
	700	64.60									
Bond-on-4 (Control group)	300										38.46
	400	38.84	39.23	39.71	40.26	40.96	41.79	42.91	44.5	46.44	48.75
	500	51.13	53.25	55.19	56.89	58.41	59.79	61.09	62.3	63.64	64.93
	600	66.15	67.20	68.02	68.52	68.72	68.53	68.21	68.0	67.88	67.77
	700	67.71									
Shade Guide	300										36.87
	400	37.30	37.72	38.18	38.63	39.16	39.75	40.63	42.0	43.95	46.27
	500	48.68	50.59	52.74	54.47	56.07	57.57	59.03	60.5	62.03	63.53
	600	64.92	66.11	67.05	67.62	67.88	67.73	67.43	67.2	67.10	67.04
	700	67.02									

(*) Indicates the statistical number allocated to each metal alloy, followed by their P values.

Table 4.6: Tristimulus Values of Dentine Firing

METAL	ILLUMINANT	X	Y	Z	x	y	Y
Ceradium V	D65	55.12	57.49	46.15	.3472	.3621	57.4881
	A	68.44	59.65	15.44	.4768	.4156	69.6497
Heranium	D65	55.69	57.99	46.19	.3483	.3628	57.9942
	A	69.22	60.26	15.46	.4777	.4157	60.2618
Remanium CD	D65	54.81	57.11	45.94	.3472	.3618	57.1116
	A	68.05	59.28	15.36	.4769	.4154	59.2759
Wirobond	D65	54.07	56.47	45.61	.3463	.3616	56.4709
	A	67.03	58.52	15.25	.4761	.4156	58.5229
Wiron 99	D65	55.51	57.75	45.80	.3490	.3631	57.7485
	A	69.11	60.06	15.33	.4783	.4156	60.0592
Bond-on-4 (Control group)	D65	56.98	58.97	45.98	.3519	.3641	58.9659
	A	71.31	61.58	15.40	.4809	.4153	61.5845
Shade guide	D65	56.10	57.52	43.85	.3563	.3653	57.5186
	A	70.71	60.47	14.68	.4848	.4146	60.4730

When comparing the non-precious base metal alloy samples, the nearest to the control group (Fig. 4.3) was Heranium, which had a reflectance range of 42.43% - 65.87% (deviations between 0.45% - 1.92%) followed by Wiron 99 which had a reflectance range of between 41.94% - 64.97% (deviation between 0.36% - 1.08%). Ceradium and Remanium had relatively similar reflectance curves with deviation ranges which were between 0.62% - 1.44%, and 0.34% - 1.63% respectively. Ceradium however, produced a slightly lower reflectance range of 42.95% - 63.87% compared with that of Remanium which was 41.82% - 64.20%. Although Wirobond produced a light oxide layer, the results of the opaque layer showed that Wirobond produced the darkest reflectance curve ranging from 41.39% - 61.96% with the deviations ranging from 1.18% - 3.34%.

4.3.2 DENTINE REFLECTANCE CURVES

During this part of the testing procedure, the shade guide was introduced into the testing process, since the shade guide matched all the other samples in terms of dimension and amount of ceramic applied.

The dentine results are presented in Table 4.5 and Figure 4.4. Table 4.5 lists the reflectance values of all five base metal alloys and the reflectance values for the gilded control group and the shade guide. Figure 4.3 is a graphic representation of the reflectance values reported in Table 4.5.

DENTINE LAYERING SHOWING PERCENTAGE REFLECTANCE

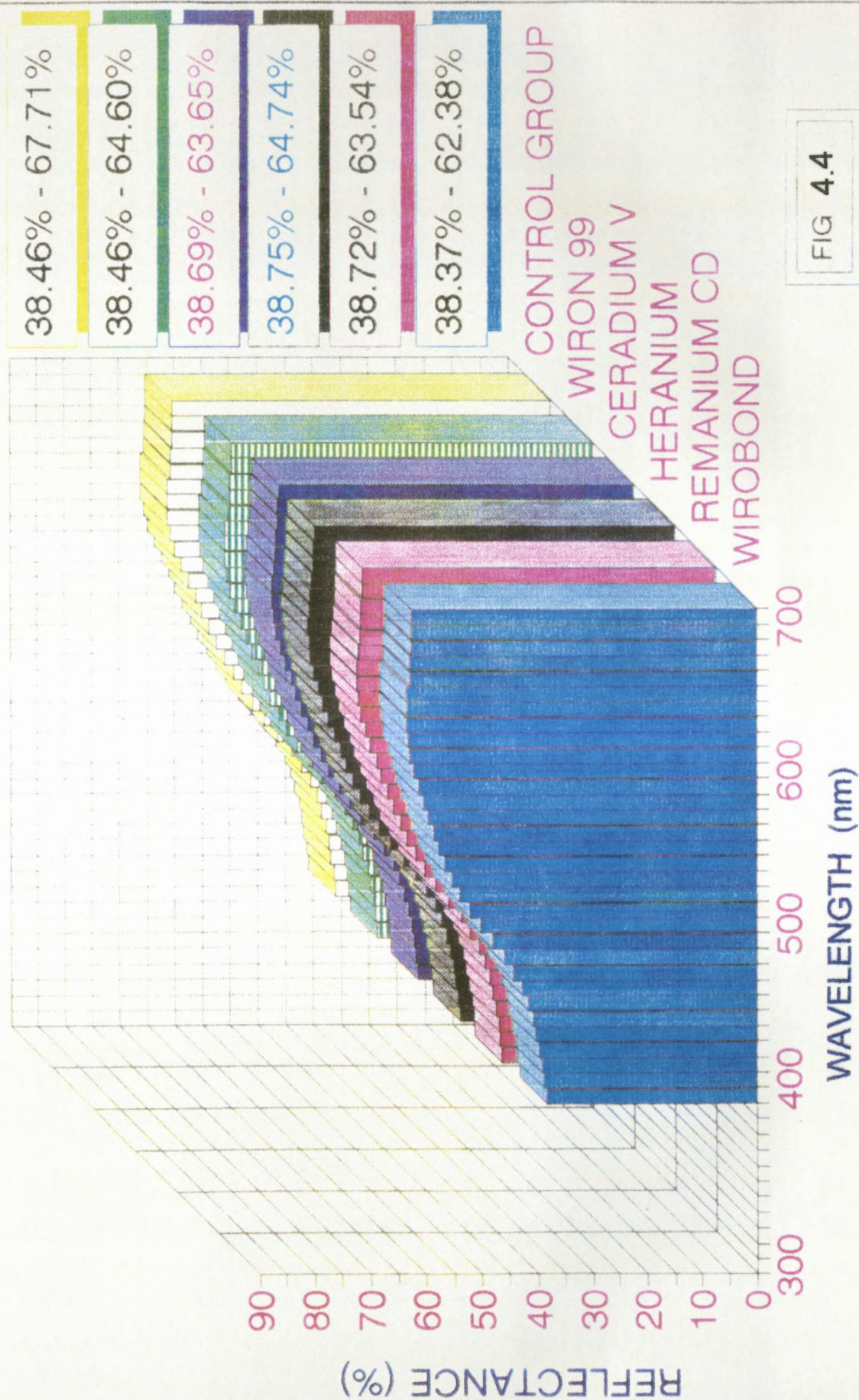


FIG 4.4

Table 4.7: Mean Reflectance Values of Glaze Firing

METAL ALLOY (*)	WAVE - LENGTH (nm)	REFLECTANCE %									
		0%	10%	20%	30%	40%	50%	60%	70%	80%	90%
Ceradium (S41) P=0.728846 ACCEPTED	300										38.20
	400	38.63	38.90	39.36	39.89	40.57	41.39	42.50	44.09	46.02	48.27
	500	50.63	52.61	54.44	56.00	57.38	58.61	59.75	60.85	61.91	62.95
	600	63.89	64.68	65.27	65.58	65.65	65.42	65.09	64.86	64.68	64.54
	700	64.89									
Heranium (S42) P=0.821134 ACCEPTED	300										38.46
	400	38.85	39.24	39.73	40.29	40.99	41.84	42.97	44.58	46.50	48.75
	500	51.04	53.06	54.87	56.42	57.78	59.00	60.12	61.20	62.23	63.25
	600	64.16	64.92	65.49	65.80	65.87	65.65	65.34	65.14	64.98	64.87
	700	64.81									
Remanium CD (S43) P=0.510156 ACCEPTED	300										37.71
	400	38.08	38.45	38.92	39.45	40.11	40.91	42.00	43.57	45.46	47.70
	500	49.96	51.95	53.74	55.26	56.59	57.79	58.89	59.95	60.94	61.92
	600	62.80	63.53	64.07	64.35	64.41	64.16	63.88	63.69	63.55	63.45
	700	63.39									
Wirobond (S44) P=0.332349 ACCEPTED	300										37.78
	400	38.15	38.52	38.98	39.50	40.16	40.95	42.01	43.54	45.37	47.51
	500	49.67	51.55	53.23	54.64	55.87	56.96	57.95	58.89	59.77	60.63
	600	61.40	62.04	62.51	62.75	62.79	62.57	62.28	62.10	61.97	61.87
	700	61.82									
Wiron 99 (S45) P=0.698088 ACCEPTED	300										38.20
	400	35.55	38.09	39.36	39.89	40.57	41.39	42.50	44.09	46.02	48.27
	500	50.57	52.61	54.44	56.00	57.38	58.61	59.75	60.85	61.91	62.95
	600	63.89	64.68	65.27	65.58	65.65	65.42	65.09	64.86	64.68	64.54
	700	64.89									
Bond-on-4 (Control group)	300										37.89
	400	38.25	38.61	39.08	39.64	40.35	41.21	42.36	43.99	45.95	48.27
	500	50.64	52.77	54.71	56.39	57.89	59.28	60.58	61.87	63.14	64.43
	600	65.63	66.68	67.50	67.99	68.19	68.00	67.67	67.46	67.30	67.21
	700	67.17									
Shade Guide	300										36.87
	400	37.30	37.72	38.18	38.63	39.16	39.75	40.63	42.09	43.95	46.27
	500	48.68	50.59	52.74	54.47	56.07	57.57	59.03	60.54	62.03	63.53
	600	64.92	66.11	67.05	67.62	67.88	67.73	67.43	67.24	67.10	67.04
	700	67.02									

(*) Indicates the statistical number allocated to each metal alloy, followed by their P values.

The dentine layer reflectance curves (Fig. 4.3) show greater uniformity among the non-precious base metal alloy samples than in the opaque layering reflectance curves. The gilded control group again showed a noticeable 3% increase in reflectance when compared with the base metal alloy samples in the 600nm - 700nm wavelength.

The gilded control group reflectance curve had a reflectance range of 38.46% - 67.71% (deviation 0.46% - 0.84%) which showed a similar reflectance range to the shade guide, which had a reflectance range of 36.87% - 67.71% and a deviation of between 0.00% - 0.00%. The reason for the low deviation displayed by the shade guide was the fact that only one specimen was manufactured and evaluated.

All the non-precious base metal alloy samples at the dentine layering stage of manufacturing had similar reflectance curves (Fig. 4.4), with only 1% or 2% differences in reflectance. Two focus ranges are reported namely: the 600nm - 700nm range and the 300nm - 400nm range. As with the opaque layering, Heranium produced reflectance curves closest to the control group, with a reflectance range of 38.69% - 64.74% (deviation range 0.41% - 1.28%). Heranium was followed by Wiron 99 which produced a reflectance range of between 38.46% - 64.60% and a deviation range of between 0.47% - 0.84%. As with the opaque layering, Ceradium had reflectance values of 38.69% - 63.65% and deviation values between 0.41% - 1.28% which were similar to the values of Remanium. Remanium had an increase in deviation around the 300 nm range (1.33% - 3.48%). This deviation however, did not affect the reflectance curves significantly, in that the reflectance curves of Remanium (38.72% - 63.54%) did not show dramatic change. The large deviation of Remanium (1.33% - 3.48) is discussed in greater detail in chapter five.

Table 4.8: Tristimulus Values of Glaze Firing

METAL	ILLUMINANT	X	Y	Z	x	y	Y
Ceradium V	D65	54.85	57.22	45.72	.3476	.3626	57.2195
	A	68.15	59.39	15.30	.4771	.4158	59.3896
Heranium	D65	55.67	57.99	46.02	.3486	.3632	57.9849
	A	69.27	60.27	15.41	.4779	.4158	60.2664
Remanium CD	D65	54.49	56.77	45.02	.3487	.3633	59.7702
	A	67.80	59.00	15.07	.4779	.4159	59.0005
Wirobond	D65	53.54	55.92	45.01	.3466	.3620	55.9239
	A	66.40	57.97	15.06	.4762	.4158	57.9683
Wiron 99	D65	55.36	57.62	45.55	.3492	.3635	57.6100
	A	68.95	59.963	15.25	.4784	.4158	59.9271
Bond-on-4 (Control group)	D65	56.48	58.46	45.38	.3523	.3646	58.4583
	A	70.73	61.07	15.20	.4811	.4155	61.0745
Shade guide	D65	56.10	57.52	43.85	.3563	.3653	57.5186
	A	70.71	60.47	14.68	.4848	.4146	60.4730

Wirobond produced low reflectance values between the focal range of 300nm - 700nm. The low reflectance of Wirobond was most noticeable in the 600nm - 700nm reflectance range with the reflectance results being as follows: 38.37% - 62.38%, and a deviation of 0.44% - 2.53%.

The nil hypothesis was accepted for all of the base metal alloys (P-values found in Table 4.5).

4.3.3 GLAZE FIRING REFLECTANCE CURVES

Table 4.7 presents the reflectance values for the final glaze firing. Figure 4.4 is a graphical representation of the reflectance values presented in Table 4.7; this Table lists the five base metal alloys, the gilded semi-precious control group and the shade guide with their related reflectance percentage data.

Upon first observation, the results of the glaze firing reflectance curves (Fig. 4.4) were relatively similar to those of the dentine layer reflectance curves. This finding might have been expected since no further ceramic was applied to the specimens during this process, and only a glaze firing was applied to all the sample groups. There were, however, significant differences in the two sets of results.

Ceradium, Remanium, Wirobond and the control group all produced a proportional drop in reflectance value of 1% or less. This drop is discussed in detail in section 4.5.

There were two exceptions to the general lowering of 1% between the dentine firing and the glaze firing, in terms of reflectance versus wavelength. Heranium, which consistently produced higher reflectance values than any of the other non-precious metals, showed an increase in reflectance around the 600nm - 700nm wavelength. This increase was from 64.74% - 64.81%. This may not be a large difference but is certainly significant compared to the other non-precious metals which showed a drop in reflectance values, resulting in the nil hypothesis being accepted for them (P-values in Table 4.7) when compared with the gilded control group. Wiron 99, which was also consistently closer to the gilded control group than the other non-precious base metal alloys, had an increase in reflectance from 64.60% - 64.89% around the 600nm - 700nm wavelength.

4.3.4 SUMMARY OF REFLECTANCE RESULTS

The major trends that were evidenced by the reflectance testing were:

- Ceradium V produced the lightest coloured oxide followed by Wirobond. Wiron 99, Remanium CD and Heranium produced similar coloured oxide layers and their reflectance values were 4% - 5% darker than those of Ceradium V and Wirobond.
- In terms of opaque reflectance, the non-precious metals produced similar reflectance curves, with the exception of Wirobond. The gilded control group proved to have a 3% - 4% higher increase in reflectance around the 600nm - 700nm wavelength than any of the non-precious metals. Wirobond produced the least reflectance around the 600nm - 700nm wavelength, which was significantly lower than that of Remanium, Heranium, Ceradium and Wiron 99. Statistically the base metal alloys were similar to the gilded control group. The nil hypothesis was therefore accepted for all the base metal alloys, with a 95% confidence level. Visually the gilded control group appeared to have a yellow hue, which reflected more profoundly than the hues of base metal alloy groups.
- The dentine reflectance curves of the base metal alloys were all similar in nature and there were no significant wavelengths of light that could have

altered the colour of any sample group, with the exception of the control group and the shade guide. The control group produced a reflectance curve which matched the shade guide and produced a 3% - 5% increase in reflectance between 600nm and 700nm. Statistically the, the nil hypothesis was accepted for the base metal alloys. It was visually observed that the gilded control group and the shade guide had an increase in the yellow / red hue.

- The glaze firing produced Y% reflectance values which were approximately 0.3% lower than the value reported for the dentine firing. This drop in reflectance, however, was over the entire spectrum (300nm - 700nm). A second set of chromaticity results presented in section 4.5 confirmed that the change in reflectance values was due to a change in Y% value (tristimulus values). Wiron 99 and Heranium were the only base metal alloys which produced an increase in reflectance around the 600nm - 700nm; however, this increase was less than 1%.

4.4 TRISTIMULUS AND CHROMATICITY RESULTS

Section 4.2 reports the results of the various base metal alloys and gilded control group in terms of percentage of light that was reflected from the specimens. In conjunction with the respective tables, the sample groups are also represented diagrammatically, thus illustrating the reflectance curves. Section 4.2 allows the reader to have a better understanding of the composition of the reflected light and where the changes of reflected light of each sample group occurred within the visible range of 300nm - 700nm.

Tristimulus values were achieved when each measured specimen was compared with three colour stimuli found in spectrophotometers (ISO/CIE 10526, 1991). Simultaneously a set of chromaticity co-ordinates were recorded (x,y). These co-ordinates were plotted on a chromaticity diagram (Annexure C) which, when plotted, produced a reference as to where and how each specimen related to the next in terms of hue.

4.4.1 INTRODUCTION TO THE CHROMATICITY DIAGRAM

The purpose of the chromaticity diagram is that it allows the reader to visualize a colour, of any sample, in a two dimensional form (hue and chroma). The third dimension (Y%) is simply the

value (or the relative amount of greyness) of the specific colour. It is important that the reader uses the chromaticity diagrams and the related bar graphs (Y% value graphs) to obtain a clear picture of the composition of the three dimensional colour discussed in the text. The hue and chroma of a specific colour can be located on the chromaticity diagram by intersecting the x and y values and by reading the bar graphs (Y%) to obtain the amount of 'greyness' in the specific colour.

Incorporated into the chromaticity diagram, are the specific wavelengths of light. These values can be located around the circumference of the coloured sphere (Annexure C). The wavelength which is of most interest to this study is the 580nm wavelength of light. The reason for the interest in the 580nm wavelength is that this is the area where most of the tooth-coloured materials are located, and the reader will be referred to this area during the general discussion in this study.

Located in the centre of the chromaticity diagram (Annexure C) is a symbol (E), which indicates the absolute white area. The specific co-ordinates are : $x=0.333$ and $y=0.333$. This is the area where all the primary and secondary colours mix to form white. It should be noted when reading the text on chromaticity values that in this study, the tooth-coloured materials were found quite close to the E value.

All the chromaticity diagrams (Figs. 4.5, 4.7, 4.9 & 4.11) represent a small portion of the actual size of the true chromaticity diagram (Annexure C). These smaller diagrams allow the reader to 'zoom' into the areas that are applicable when reading the chromaticity co-ordinates. The colours found around the edge of the diagram are not proportional to the actual intensity of colour represented on the true chromaticity. The border colours do however represent the area of colour found around the true chromaticity diagram. These border colours should then only serve as references as to which direction (in terms of colour) the specimen might be 'moving', to or from, from one firing layer to the next. By using the chromaticity diagrams one can compare the position of one sample group with the next, and hence establish whether that specific sample group was closer to one area of colour than the next sample group.

OXIDE FIRING

CHROMATICITY COORDINATES

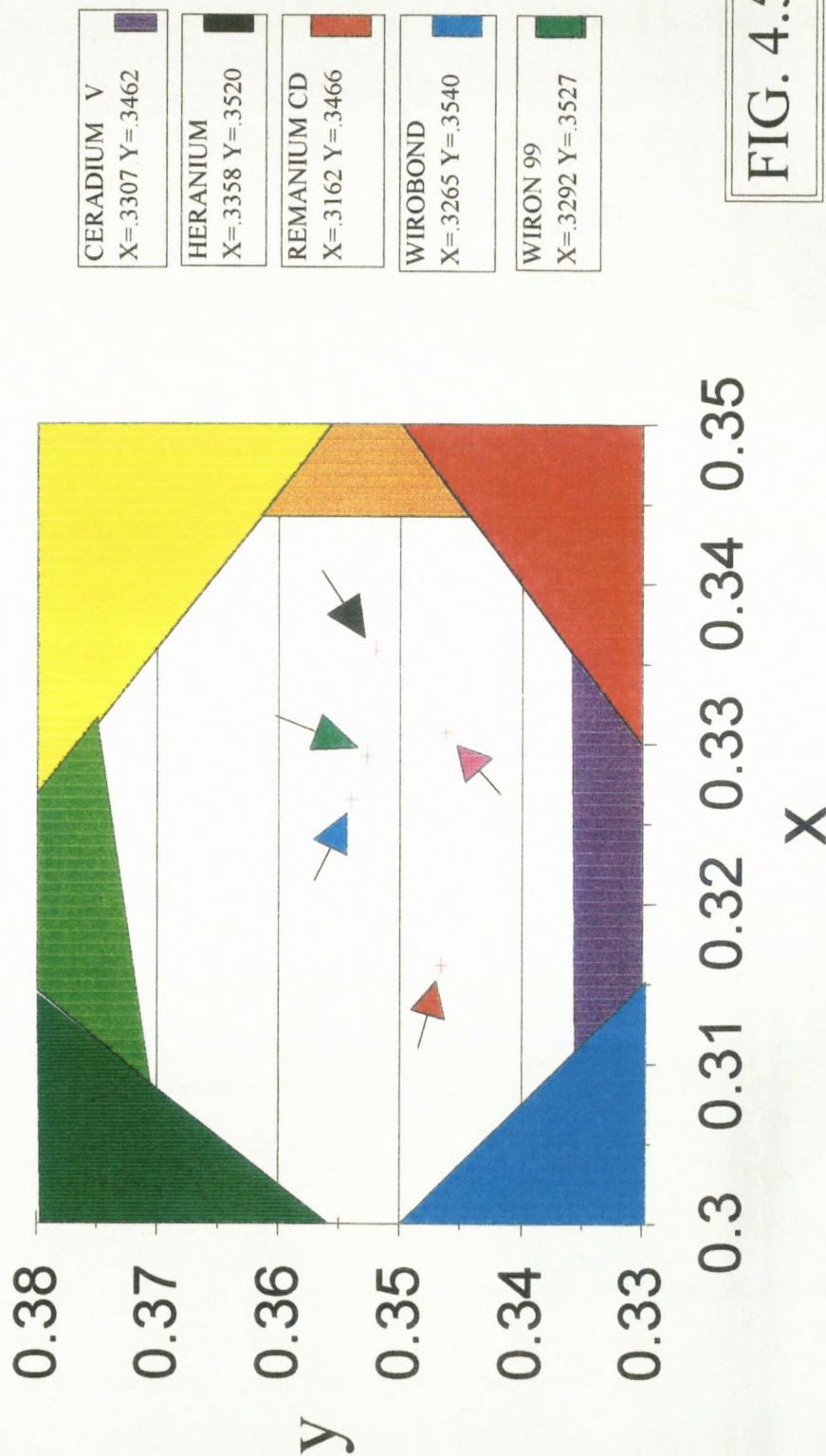
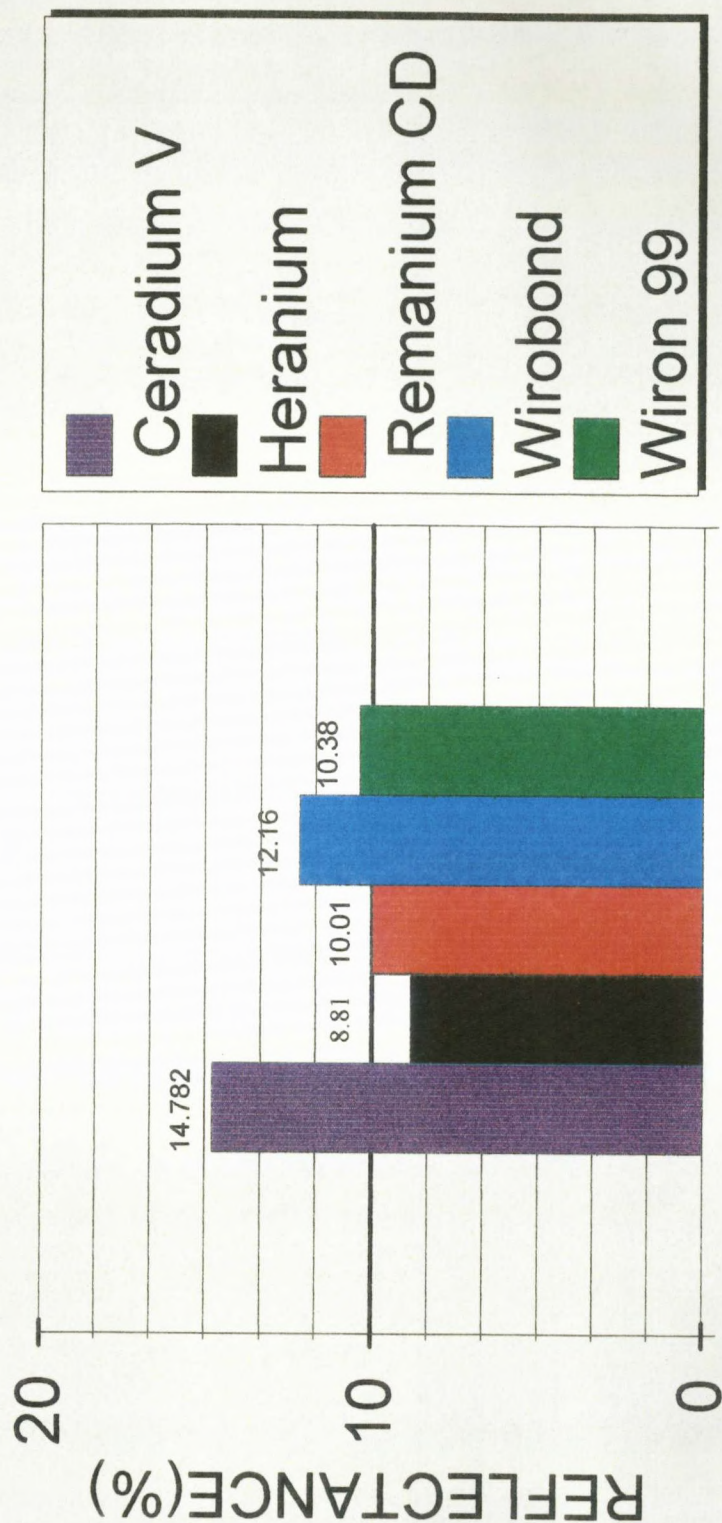


FIG. 4.5

OXIDE FIRING

Y% (VALUE) SPECTROPHOTOMETER READINGS



BASE METAL ALLOYS

FIG 4.6

4.4.2 THE OPAQUE FIRING

Table 4.4 lists the two illuminates that were used for testing the tristimulus values: D65 is a natural light and A is an artificial light source. Since the D65 light source is the recommended light source for colourmetric testing (ISO/CIE 10526, 1991), the focus of the tristimulus and chromaticity values was read under D65 lighting conditions.

Base metal alloys falling within 0.01 linear unit (one colour difference unit [Fig. 4.7]) of one another on the chromaticity diagram were considered insignificant (Crispen *et al.*, 1991).

Ceradium and Wirobond, although producing light oxide firing colours (Fig. 4.6), reflected low chromaticity values (Table 4.4). Ceradium's chromaticity values ($x=0.3396$; $y=0.3573$) were slightly lower than those of Wirobond ($x=0.3395$; $y=0.3576$), although the differences were very slight. Ceradium produced a reflectance value which inclined more towards the 400nm wavelength (purple light) than that of Wirobond.

Heranium, Remanium and Wiron 99 were also grouped together on the chromaticity diagram (Fig. 4.7) and show a larger increase in orange and yellow hues than Ceradium and Wirobond. Wiron 99 ($x=0.3423$; $y=0.3593$) was located nearest to the gilded control group followed by Heranium ($x=0.3419$; $y=0.3591$) and then Remanium CD ($x=0.3415$; $y=0.3590$).

The distribution of the base metal alloys on the chromaticity diagram was grouped within 0.01 linear units, which is the accepted tolerance for the distribution of sample groups on the chromaticity diagram. The gilded control group was located more than 0.005 linear units from the base metal alloys, therefore the base metal alloys were considered to be insignificantly different from one another, but significantly different from the gilded control group.

The gilded control group ($x=0.3462$; $y=0.3610$) reflected an increase in hue and chroma significantly greater (0.005 linear units) than any of the non-precious metal alloys and tended more towards the 580nm wavelength (natural tooth colours). The shade guide ($x=0.3550$; $y=0.3648$) produced a result which was significantly greater than that of the non-precious alloys and displayed chromaticity values which were closer to the 580nm wavelength than those of the gilded control group.

OPAQUE FIRING

CHROMATICITY COORDINATES

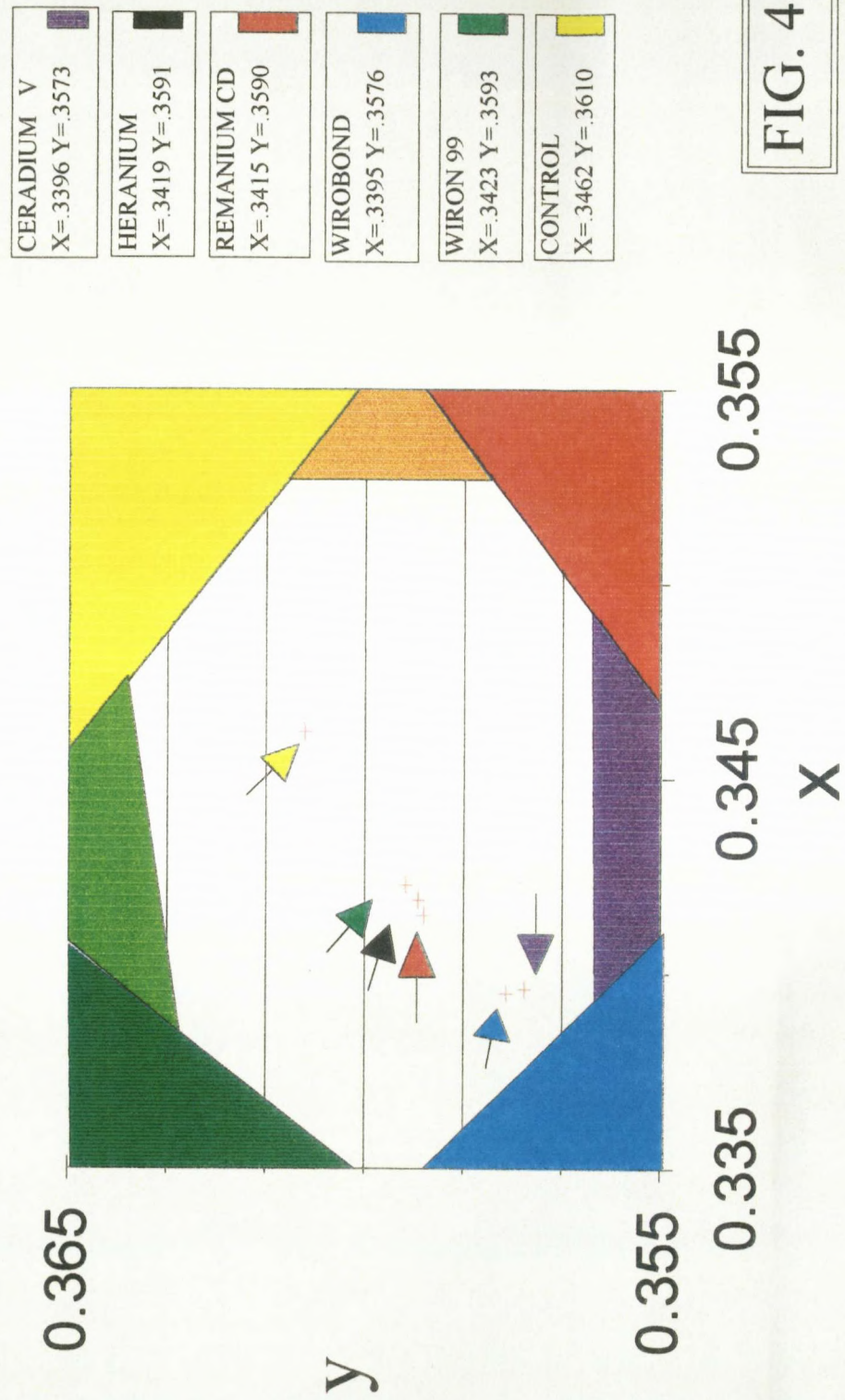
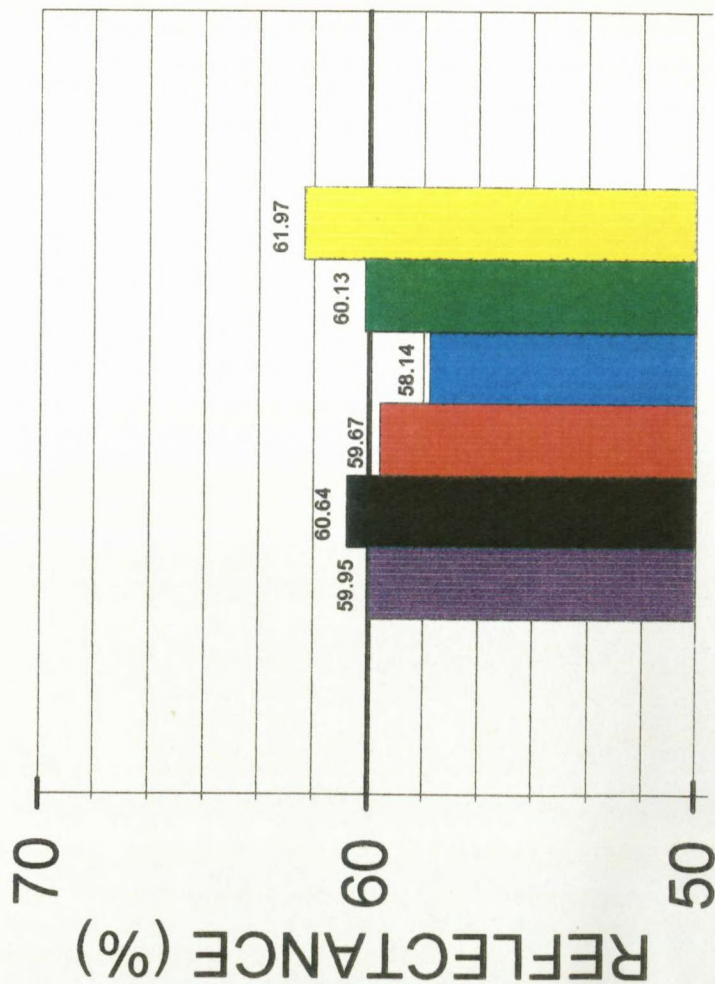


FIG. 4.7

OPAQUE FIRING

Y% (VALUE) SPECTROPHOTOMETER READINGS



BASE METAL ALLOYS

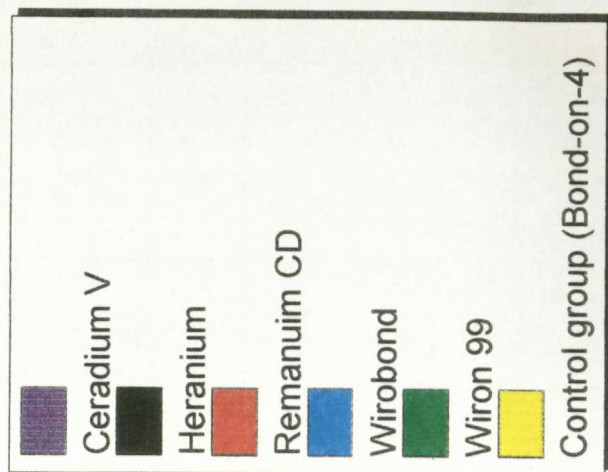


FIG 4.8

4.4.3 THE DENTINE LAYER

The results of the dentine firing (Fig. 4.7, Table 4.6) showed a correlation with the opaque tristimulus values in that there was proportional increase in hue and chroma (x,y) and a proportional decrease in value ($Y\%$).

Wirobond chromaticity values ($x=0.3463$; $y=0.3616$) reflected hues which were green in nature and darker ($Y=56.47\%$) than Remanium ($x=0.3472$; $y=0.3618$; $Y=57.11\%$) and Ceradium ($x=0.3472$; $y=0.3621$; $Y=57.49\%$). Heranium showed an increase in yellow chroma ($x=0.3483$; $y=0.3628$) when compared with Ceradium. Of the non-precious base metal alloys, Wiron 99 produced the best results, with chromaticity values of $x = 0.3490$ and $y = 0.3631$. Although Wiron 99 produced a $Y\%$ value of 57.75% which was lower than that of Heranium ($Y=57.99\%$), Wiron 99 showed important tooth-coloured hues (Fig. 4.7) that were closer to the gilded control group than those of Heranium.

The gilded control group, in terms of chroma, moved closer to the shade guide and reflected tristimulus values of $x= 0.3472$ and $y=0.3620$.

The shade guide remained closer to the 580nm wavelength than any of the sample groups with reflectance values of $x= 0.3550$ and $y = 0.3648$.

The shade guide and the gilded control group remained significantly different from the base metal alloy samples with the gilded control group and the shade guide remaining at least 0.01 linear units away from the base metal alloys (Fig. 4.9).

Figure 4.10 shows the $Y\%$ tristimulus values of the dentine layer. These results show an interesting relationship between hue, value and chroma when compared with the results presented in Figure 4.9. It can be noted that although the x,y chromaticity values of the shade guide reflected hue and chroma which were superior to those of the base metal alloys, the shade guide displayed a lower $Y\%$ value when compared with the $Y\%$ values of the base metal alloys. The reason for this is fully discussed in chapter five.

DENTINE FIRING

CHROMATICITY COORDINATES

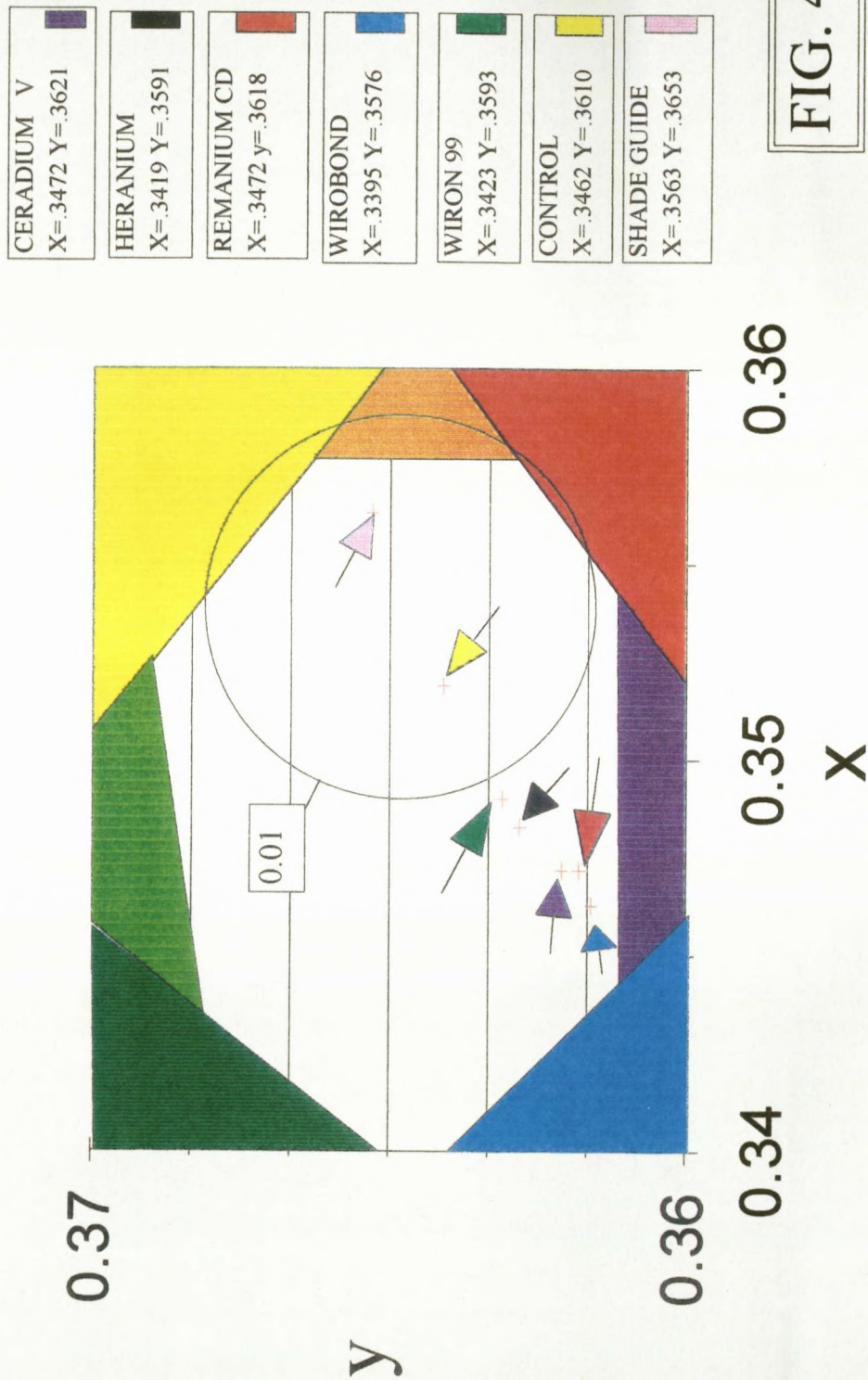


FIG. 4.9

DENTINE FIRING

Y% (VALUE) SPECTROPHOTOMETER READINGS

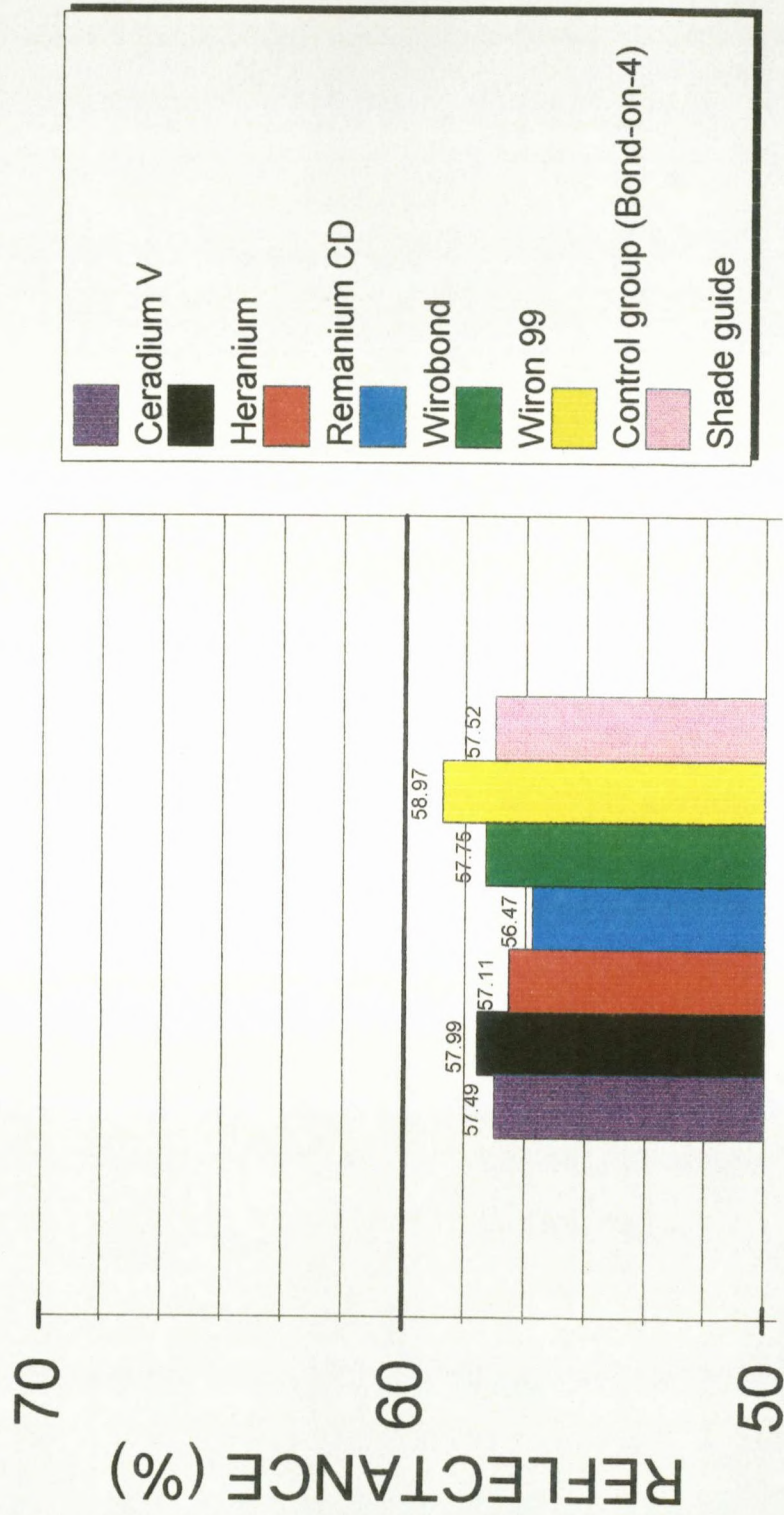


FIG 4.10

BASE METAL ALLOYS

GLAZE FIRING

CHROMATICITY COORDINATES

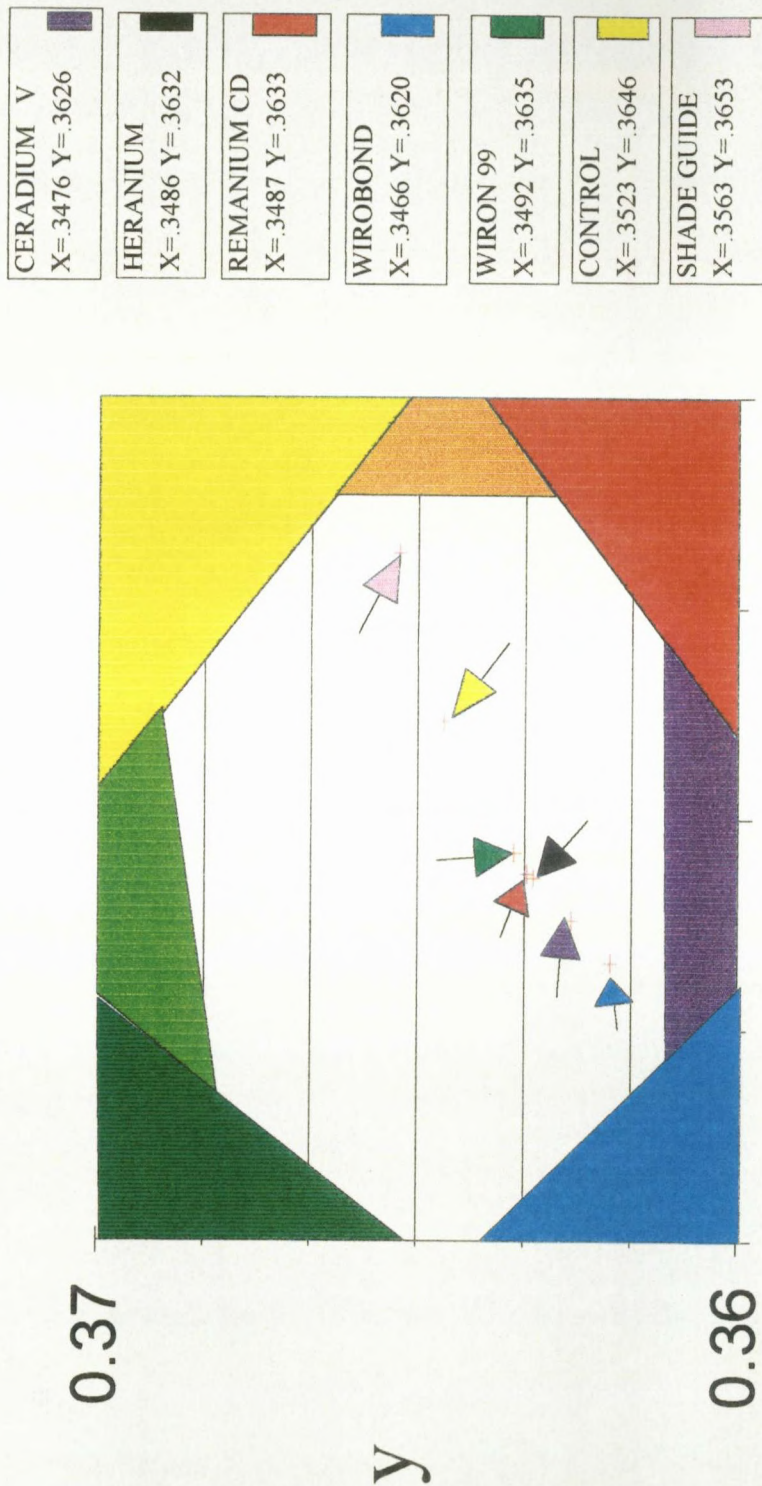
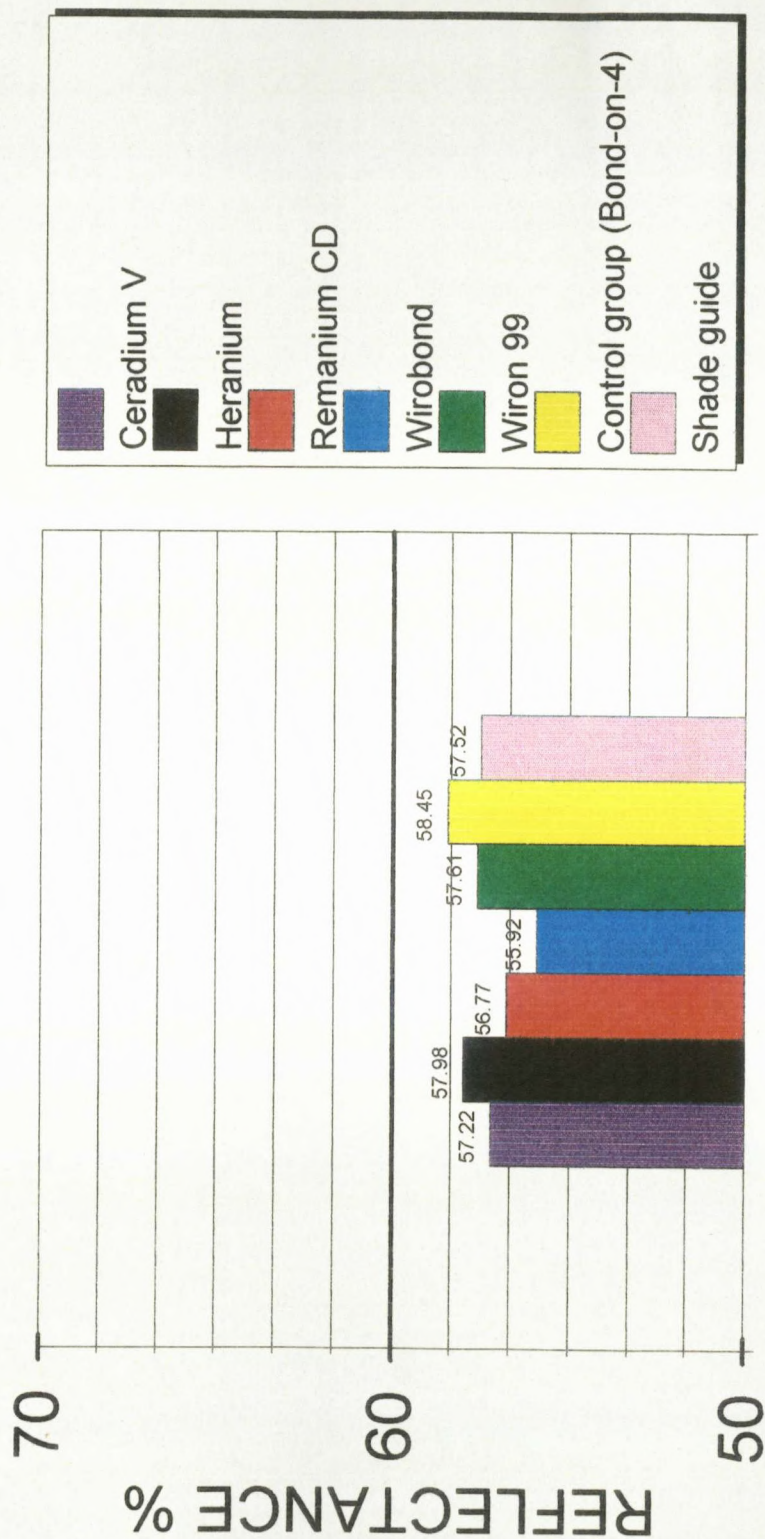


FIG. 4.11

GLAZE FIRING

%Y (VALUE) SPECTROPHOTOMETER READINGS



METAL ALLOYS

FIG. 4.12

4.4.4 DENTINE VERSUS GLAZE FIRING

All the sample groups showed a drop in reflectance value from the dentine firing to the glaze firing. It was also observed that the 0.3% drop in the Y% value was consistent in most sample groups, with the exception of Wiron 99 and Heranium, which showed a smaller but similar (i.e. 0.02%) decrease in value. This proportional decrease suggests that the difference was caused by changes in the grain boundary structure, which is discussed in greater detail in chapter five.

4.4.5 SUMMARY OF THE CHROMATICITY RESULTS

The major trends that were evidenced by the chromaticity testing were:

- The chromaticity co-ordinates of the oxide firing (Fig. 4.5) displayed significant differences in the colours of the base metal alloy oxides.
- The opaque firing (Fig. 4.7) results showed that Ceradium and Wirobond produced similar reflectance values in terms of hue and chroma; i.e. they were lighter in colour than the rest of the base metal alloys because they tended to be closer to the absolute white value ($E = 0.333; 0.333$)(Annexure C). Heranium, Remanium and Wiron 99 showed a greater tendency to reflect chromaticity positions that were closer to the 580nm wavelength (tooth-coloured light). The gilded control group was significantly closer to the 580nm wavelength (increase in yellow chroma) than the non-precious metal alloys. The shade guide was closer to the 580nm wavelength than the gilded control group.
- The dentine firing results (Fig. 4.4) correlated with the opaque results; however, after the dentine firing process there was a general increase in hue and chroma of all the sample groups towards the 600nm - 700nm wavelength.
- The gilded control group produced the lightest Y% reflectance results (Fig. 4.10), followed by Heranium, Wiron 99, Ceradium V, Remanium CD, and Wirobond, which was the darkest.
- The Y% dentine reflectance values (Fig. 4.10) showed that although the shade guide had a favourable hue and chroma, in terms of value (Y%) the results revealed that the shade guide was darker than most of the non-precious metals, with the exception of Wirobond.

4.5 LIGHT MICROSCOPE RESULTS

Description of end-on sections of specimens in Annexure A

The results of the light microscope analysis (200x magnification) displayed no excessive oxide penetration into the opaque layering. Wirobond showed a small amount of oxide penetration. However when viewing the reflectance curves of the opaque layering (Fig. 4.3) it was concluded that there was no significant influence from this oxide layer.

The results of the gilded layer (X) showed a thick gold foundation which was responsible for the increase in hue and reflectance value of the sample group. The rough reflective surface of this gilded layer was also an influencing factor in the reflectivity of the sample group. The light microscope results are discussed in greater detail in chapter five.

4.6 VISUAL ANALYSIS

NOTE: After the analyses and evaluation of the objective results, it was argued that the integrity of the study could only be enhanced by including a subjective (i.e. visual evaluation) of the specimens as well (Billmeyer and Saltzman, 1981: 67 - 109). This is discussed in detail in Chapter five.

CHAPTER FIVE

DISCUSSION

5.1 INTRODUCTION

The goal of this study was to establish the relationship between five selected base metal alloys, and a custom made shade guide by objectively measuring the colour of five selected base metal alloy groups after firing and then comparing these colours with that of the custom made shade guide using a reflectance spectrophotometer. Subsequently, the specimens were also subjected to a subjective analysis (i.e. visual evaluation of each specimen).

The main findings were:

- The oxide colours of the five selected base metal alloys showed a 2% - 4% difference in Y% reflectance value reflectance (Fig. 4.6).
- The chromaticity diagrams (Fig. 4.7 & 4.9) show that the custom made shade guide reflected more natural tooth colours than the five selected base metal alloys.

5.2 SUBPROBLEM ONE

The hypothesis for subproblem one, namely that a relationship existed between base metal alloy oxides and that the differences in colour could be demonstrated, was accepted on the following bases:

- Through the use of a spectrophotometer, it was possible to establish differences in the oxide colours of the base metal alloys.
- The reflectance curves of the oxide firing results showed significant differences among the base metal alloy sample groups.

A discussion on the reasons for accepting the validity of these two findings follows:

5.2.1 SPECTROPHOTOMETER TESTING

By using a spectrophotometer, measurable, accurate and valid results were produced for the evaluation of metal-ceramic porcelain. The accuracy of the spectrophotometer was demonstrated by the fact that it was possible to record very small deviations within the base metal alloy specimens' colour reflectance data (0.17% - 0.37%), as well as significant differences between the base metal samples after the testing of the oxide layer. The differences in the reflectance values (5%) of the base metal alloy sample groups after testing proved to be significant, which resulted in the rejection of the nil hypothesis with a 95% confidence level (Table 4.1). The value of the spectrophotometer as an instrument for the measurement of colour was further confirmed when the reflectance results of the opaque and dentine firing processes (Figs. 4.2 and 4.3) of the base metal alloy groups revealed insignificant differences between one sample group and the next. By applying the two-way analysis test, the nil hypothesis was accepted with an 0.05 degree of freedom. Such objective distinctions could not have been possible with the human eye.

Yamamoto (1992:9-36) who also used spectrophotometer readings to compare different tooth colour shades, is a reputable researcher in this field of ceramic technology. Although Yamamoto's study measured different shades and varying thicknesses of ceramic, the results of his study confirmed the use of objective evaluation metal-ceramic colourimetry and the use of the spectrophotometer.

In a review of the literature, it was noted that research methods tend to utilise only one method of colourimetric analysis, for example: spectrophotometer (Lund *et al.*, 1985) or the chromascan (Goodkind *et al.*, 1985). The results of these research approaches, although valid, focus on one aspect of colour shade evaluation only, while the most important tool in metal-ceramic colourimetry, namely visual analysis, has not been used to its full extent in the process of trying to achieve extreme accuracy.

5.2.2 VISUAL ANALYSIS

Billmeyer and Saltzman (1981:67-87) recommended the use of visual analysis during the final stages of any colourimetric analysis. Billmeyer and associate argue that visual analysis can be used in conjunction with objective instrumentation to confirm colour reproducibility of the final

restoration process. Due to the fact that ceramic restorations are ultimately viewed by the human eye, Billmeyer and Saltzman reinforced the use of a final visual analysis, irrespective of whether the results of the computed statistics and the visual result agreed or disagreed.

Therefore, the ensuing discussion focuses not only on computed results and findings but also on findings obtained by evaluating each specimen by visual analysis. This was done in an attempt to ensure a comprehensive and inclusive comment on the colour of the specimens evaluated in this study.

Although objective instrumentation is neither cost nor time effective in the commercial dental laboratory, a better understanding of the effects any base metal alloy oxide has on metal-ceramic tooth colour shades would greatly enhance the production of the final product.

5.2.3. REFLECTANCE CURVES OF THE BASE METAL OXIDES

The second set of factors which contributed to the acceptance of hypothesis one was the impact of metal oxides produced by the oxidation of selected base metal elements found in the composition of the five base metal alloy groups. The base metal alloys used in this study fell into two broad categories: beryllium containing and non-beryllium containing.

Table 5.1: Basic Composition of Base Metal Alloys

BERYLLIUM CONTAINING	NON-BERYLLIUM CONTAINING
Ceradium V (containing less than 2% Beryllium)	Wirobond (cobalt-chrome) (less than 68% cobalt, chromium 25%- 27%) Heranium Remanium CD Wiron 99 (nickel-chrome) (Less than 80% nickel, 13%-22% chromium and 0% beryllium)

Williams (1990:42) reports that beryllium containing base metal alloys produce a light oxide as opposed to the non-beryllium alloys, which produce darker and thicker oxides. In the restoration

process a light coloured oxide is desirable. However, Phillips (1982:559) indicates that the inhalation of beryllium is potentially toxic under certain uncontrolled conditions; consequently a conflict exists between the usage of beryllium containing base metal alloys and the non-beryllium containing base metal alloys. What is of importance is that the potentially carcinogenic beryllium element that the commercial technician is faced with, forces him/her to make an appropriate selection.

5.2.3.1. BERYLLIUM CONTAINING BASE METAL ALLOYS

The results showed that the reflectance curve of Ceradium V (Fig. 4.2) produced the lightest coloured oxide of all the base metal alloys.

Phillips (1982:548) noted that iron, copper and beryllium were added to alloys to reduce fusion temperature. Beryllium is noted for its ability to reduce excessive oxide formation (Williams, 1990:42) along with manganese and silicon. It was for this reason that it was expected that Ceradium V, a beryllium containing base metal alloy, would produce lighter coloured oxides, followed by the non-beryllium containing base metal alloys, namely the nickel-chrome and cobalt-chrome alloys.

Phillips (1982:548) and Williams (1990:42) commented on the role of beryllium in the regulation of the oxide formation of base metal alloys, and the results of their studies confirmed speculations about the lighter coloured oxide colour of Ceradium V. In the present study, it was also noted that the reflectance curve showed no fluctuations of colour between 300nm and 700nm. By analysing this focal range of visible light reflected off the specimens, it was possible to notice any increases within the 300nm - 700nm range. These areas, or fluctuations, of reflected wavelength enable the detection of the dominate of the specimen. It was concluded that the beryllium containing alloy may have had a stabilizing effect on the oxide formation, in that there was no fluctuations of colour emerging during oxide reflectance testing of Ceradium V. When viewing the reflectance curve of Ceradium V (Fig. 4.2), it was noticeable that the reflectance increased uniformly, displaying a classic achromatic colour (Yamamoto, 1985:228). Similar findings were reported by Yamamoto (1985:228) in that achromatic colours were produced when there was a uniform change in reflection and absorption, or when a colour was produced by the mixing of pure black and white. When relating the strength of the Ceradium V curve to the rest of the base metal alloys, it was noticed that the base metal alloy curves showed fluctuations in reflection and

absorption as opposed to the uniform nature of Ceradium V (Fig. 4.2). Yamamoto recognised the nature of these fluctuations as chromatic colours. It must be noted that although the word chromatic colour indicates that the sample has chroma (colour), it is fully understood that the oxide colours are grey. Yamamoto states that it is the specific amounts of reflected and absorbed light that are mixed to produce various colours. It is the researcher's opinion that the fluctuations in reflectance between 300nm and 700nm of the remaining four base metal alloys (Wirobond, Remanium, Heranium and Wiron 99) (Fig. 4.2) may have been a result of chromatic colour mixing.

Figure 4.6 provides information on the Y% or value of the base metal alloy colours. Ceradium V showed a stronger reflectance of 2.6% over and above Wirobond. Having a high Y% value of 14.782%, it was speculated that Ceradium V would produce the best results when the ceramic layer was tested. The overall reflectance of the oxide colour indicates that the oxide colour of Ceradium V was lighter than the colour of any of the other base metal alloys tested in this study.

5.2.3.2. NON-BERYLLIUM CONTAINING BASE METAL ALLOYS

The non-beryllium containing base metal alloys used in this study were (Table 5.1):

- Wiron 99
- Heranium
- Remanium CD
- Wirobond

It was hypothesised that the non-beryllium containing base metal alloys would not perform as well as the beryllium containing base metal alloys in terms of oxide colour.

Wirobond being nickel and beryllium free, contains between 25% - 27% chromium to increase the oxidation resistance of the base metal alloy. It was therefore speculated that Wirobond would produce a dark coloured oxide layer and that, due to its high chromium content, this oxide layer would be stable in nature and have little or no effect on the colour of the applied ceramic.

Wirobond was the only cobalt-chrome base metal alloy tested in this study to produce a reflectance curve (Fig. 4.2) which was substantially weaker (2.6% with this lower value displaying a darker colour) in the reflectance values between 300nm - 700nm when

compared with the reflectance values of Ceradium V. The Y% values (Fig. 4.6) of the Wirobond oxide firing showed an interesting result. As stated previously in this section it was speculated that Wirobond would produce a dark coloured oxide layer. Although the Y% value of Wirobond (Fig. 4.6) was darker than that of Ceradium V, it produced a Y% value which was 2% - 4% lighter than Heranium, Remanium CD and Wiron 99. It is suggested that the high chromium content of Wirobond (25% - 27%) may have produced a greater degree of oxidation resistance than originally speculated.

Strandman (1982:67) noted that when the chromium concentration of cobalt-chrome alloys was 25%, the oxidation resistance was at its maximum. He further stated that the amount of oxide formed on cobalt-chrome alloys was small and easily removable. Strandman also noted that the oxide layer consisted of two separate layers. The first layer forming the bond between the metal alloy and the oxide, was white, and this internal layer was then covered by a second external layer which was black. There was no indication by Strandman as to the deleterious effects of this dark oxide layer on the resultant shade of the ceramic.

It was observed that the nature of the Wirobond reflectance curve (Fig. 4.2) was different from the curves of Ceradium and Wiron 99. The Wirobond curve showed an increase in the reflectance range of 500nm - 600nm. The increase in this range indicates that the colour of the oxide reflected a larger percentage of the green wavelength (500nm) and absorbed blue (450nm) and orange (600nm).

Although the Wirobond oxide colour reflected three different colours, it should be noted that when red, green and blue are additively mixed, the colour as observed by the human eye is white. Therefore, theoretically the resultant colour of the oxide colour should be light grey, because of the low Y% value of Wirobond (remembering that Y%= amount of greyness). The latter was supported visually (Fig. 3.9 [C]) and the oxide colour of Wirobond compared well with the colour of Ceradium. The difference between Ceradium and Wirobond however lay in the constitution of the reflected colour. Ceradium had a reflectance which was mono-chromatic in nature and therefore produced a mono-chromatic grey colour as opposed to the chromatic mixture of red, green and blue of Wirobond.

The emergence of these different reflected colours produced by Wirobond is not fully understood. However, the results also confirmed the shape and nature of Yamamoto's (1985:234) reflectance curves of achromatic and chromatic colours. Yamamoto

(1985:276) reported the use of metallic oxides for the pigmentation of ceramic. He showed that metal oxides used for the pigmentation of ceramic were the following:

- cobalt oxide = blue
- chromium = green
- complexes of manganese or iron = pink/red

In Figure 4.2 the reflectance curve of Wirobond shows a fluctuation between the 500nm - 600nm wavelengths. Since green (500nm), blue (400nm) and pink (680nm) were the ranges which reflected increases and the fact that cobalt and chrome are found in the composition of Wirobond, may substantiate the reason for the reflectance of these specific wavelengths of colour.

5.2.3.3. NICKEL- CHROME METAL ALLOYS

The reflectance curves of the nickel-chrome base metal alloys (Remanium CD, Heranium and Wiron 99) (Fig.4.2), showed that the reflectance values between the focal ranges of 300nm - 700nm displayed similar percentages of reflectance.

Like Wirobond, Remanium CD showed an increase in reflection between the 500nm and 600nm range. Again the fluctuation in the 500nm region showed an increase in the reflectance of the green wavelengths of light. As mentioned in section 5.2.3.2, the reflectance of green in the 500nm wavelength may have indicated the presence of chromium oxides, and since Remanium shows a 13% - 22% chromium content in its composition, this may well have been the reason for the increase in the 500nm (green) reflectance region.

Although Heranium, Remanium and Wiron 99 were similar in colour, all of these base metal alloys reflected, in general, a 3% lower percentage of hue and chroma throughout the focal range of 400nm -700nm than Ceradium V and Wirobond. This was expected due to the metal compositions being similar in nature and therefore producing similar oxide formations. The Y% values in Figure 4.6 show that the nickel-chrome metal alloys were darker (lower Y% values) than the cobalt-chrome and beryllium containing alloys. The reason for the low Y% value of nickel-chrome alloys is not entirely clear: however, Baran *et al.* (1983:639) noted that the rare earth elements such as tin, control the oxidation of the nickel-chrome alloys and that the cobalt-chrome alloys are significantly more noble than the nickel-chrome alloys. The presence of these earth metals in nickel-chrome alloys and

the fact that they are less noble than the cobalt-chrome metal alloys may suggest that the nickel chrome alloys had less oxidation resistance than cobalt-chrome, and therefore produced a darker oxide.

What should be of value to the dental technician is the knowledge of how the base metal elements in the metal alloys affect the colours of the oxide layers. Therefore when choices are made as to which brand of metal-ceramic alloy is to be used, then the dental technician will be able to make an appropriate choice. It should be borne in mind that, commercially, the nickel-chrome alloys are the most popular of the base metal alloys, mostly due to their availability and cost effectiveness (Table 3.1). However, in this study the overall oxide colour of all the nickel-chrome base metal alloys was darker than that of the cobalt-chrome and the beryllium containing base metal alloys (Fig. 4.2).

A second noticeable factor in the reflectance patterns of the nickel-chrome base metal alloys was that the reflectance curves of Wiron 99 and Heranium increased proportionally between the focal ranges of 300nm - 700nm as opposed to the reflectance curve of Wirobond which had a 5% increase in reflectance around the 550nm range. Remanium CD did display slight fluctuations around the 550nm wavelength, but these fluctuations were not as noticeable as those observed for Wirobond.

The importance of visual analysis cannot be underscored enough, because similar methods will be employed in the commercial dental laboratory, when the acceptance criteria are being viewed. The question is whether the commercial dental technician would apply the criteria when visual analysis is used. Visually (Fig. 3.9) the base metal alloy colours of the nickel-chrome sample groups (B, D and E) were darker than the rest of the base metal alloy sample groups. The oxide formation of the nickel-chrome alloys also appeared to be thicker with a more uniform coverage of the metal surface than the beryllium and cobalt containing base metal alloys. At that stage, based on the literature (Strandman 1982:67) it was predicted that due to the darker colour of the nickel-chrome base metal alloys, the effects of the oxides on the ceramic layers would be more significant than those produced by the beryllium and cobalt containing base metal alloys.

NOTE : After the firing of the base metal alloy oxides, there was evidence of a light 'halo' around the circumference of some of the disc shape specimens. This phenomenon was noted specifically after the firing cycle of the Ceradium V base metal alloy group

(Fig. 3.2.7 -A). The 'halo' found on the Ceradium V specimens was noted prior to the opaque firing. Furthermore, no reports of similar findings were found in the literature. The investigation of this phenomenon was beyond the scope of this study and may be a topic for further research in the future.

5.3 SUBPROBLEM TWO

In order to achieve the goal of comparing the different shades of the five base metal-ceramic alloy groups after the application of ceramic, it was important to establish the colour of each individual ceramic layer. In this context it was hypothesised that the type of relationship between the shade of porcelain after each individual layering of ceramic and the base metal alloy groups would affect the final colour of the metal-ceramic restoration. Four factors were considered before the hypothesis could be accepted or rejected:

- **Y% value:** In terms of the opaque layering, the Y% value of the cobalt-chrome alloy (Wirobond) was almost 2% lower than that of the rest of the base metal alloys (Remanium CD, Wiron 99, Heranium, Ceradium). Statistically, this difference proved to be significantly low.
- **Hue, value and chroma:** The opaque layering results showed that the base metal alloys produced significantly different result, in terms of hue, value and chroma when compared with the gilded control group and the shade guide.
- **The performance of opaque, dentine and glaze results:** when plotted on the chromaticity diagrams, the opaque, dentine and glaze results as presented in Chapter four show that all the base metal alloys reflected insignificant differences when compared with one another.
- **Reflectance diagrams of the opaque, dentine and glaze firings:** The reflectance diagrams in Chapter four show that the opaque, dentine and glaze firings resulted in insignificant differences among the colour compositions of the base metal alloys

1. Y% VALUES

Wirobond reflected a Y% of 58.14% (Fig. 4.8) after testing the opaque layering. This value was almost 2% lower than that of the remaining base metal alloys, showing that Wirobond was darker than Remanium, Ceradium V, Heranium and Wiron 99. Although the statistical P - values presented in Table 4.2 indicate that Wirobond's opaque layer was significantly

similar ($P=0.148489$) to the rest of the base metal alloys, the visual analysis confirmed that Wirobond's opaque layer was indeed darker than the rest of the base metal alloy opaque layers.

When a cross sectional specimen was viewed under 200x magnification (Annexure C), it was noted that Wirobond's oxide layer was slightly more prominent than that of the rest of the sample groups. The instrumentation used did not allow the exact measurement of this oxide layer. However, visually, the oxide layer of Wirobond appeared to be thicker. The thicker oxide layer of Wirobond is not a confirmed result, and the darker coloured opaque layer of Wirobond may suggest that the visually thicker oxide layer might have had an effect on the resultant colour of Wirobond's opaque colour. Further research is required into the thickness of Wirobond's oxide layer after the firing of ceramics.

Ceradium V, a beryllium containing base metal alloy, produced an oxide colour which proved to have a significantly lighter coloured oxide in terms of hue, value and chroma when tested at the oxide layering stage (section 5.2.3.1). However, when tested after the application of the opaque (Fig. 4.7) it showed a move away from the natural warm colour of the gilded control group and the rest of the base metal alloys. It was suspected that Ceradium V would produce a result that showed a close similarity with the gilded control group because of the light coloured oxide; however, the reason for the poor performance of Ceradium V is not known. It is the researcher's opinion that the opaque layer may in fact have performed according to the requirements of masking out the undesirable colour of the dark underlying oxide colour.

2. HUE, VALUE AND CHROMA

It was observed that:

- The base metal alloys (Fig. 4.7) were grouped into a 0.01 unit of colour difference away from the gilded control group.
- The base metal alloys all showed a lower Y% value than the gilded control group (Fig. 4.8).

After the opaque firing, all of the base metal alloys fell within 0.01 linear unit of one another (Fig. 4.7). The gilded control group on the other hand was located outside this focal range.

Similar observations were made by Crispen *et al.* (1991:355) when they noted that specimens falling within one colour difference unit were considered significantly similar. This suggests that, although the masking abilities of the opaque layer were consistent, the final colour of the base metal alloy opaque was not the natural yellow tooth-coloured hue, as that produced by the gilded control group. The difference in colour between the base metal alloys and the gilded control group was significant, as could be seen with the naked eye.

The reason for the differences in colour when comparing the gilded control group and the base metal alloys becomes obvious when the light microscope results (Annexure A [X]) are viewed. When viewed under 200x magnification, the gilded control group showed a distinctive gold coloured layer.

A question to which the answer was of utmost importance, was why the gilded control group enhanced the warm colour of the gilding material through the opaque layer, whereas the base metal alloys showed no comparative differences in colour, irrespective of the fact that they too had different coloured underlying colours. Crispen *et al.* (1991:356) found that the type of metal alloy used could affect the resultant shade of the ceramic restoration. Although Crispen and his associate tested different metal alloys, their conclusions were supported by the findings of this study. The fact that the base metal alloys all had a similar hue (dark grey) and the gilded control group a golden hue, suggests that the hue of the metal foundation was the determining factor affecting the resultant colour of the final restoration. The importance of this finding for the dental technician in the commercial dental laboratory must therefore be emphasized.

3. THE PERFORMANCE OF THE OPAQUE, DENTINE AND GLAZE FIRINGS

Firstly, with reference to reflectance patterns (Fig. 4.4), it was noticed that after the dentine firing the base metal alloys all appeared to perform in a similar pattern.

The results confirmed that two factors namely:

- methodology (instrumentation i.e. : depth gauge)
- amount of applied ceramic

confirmed and validated the methodology used in this study for the ceramic application.

The dentine layers of the sample groups produced an increase in hue and chroma towards the yellow wavelengths of light, which was predictable due to the colouring of the applied ceramic.

The second factor influencing the colouring of the dentine layer was the amount of ceramic applied. In this investigation the depth of the ceramic was uniform (1mm) and conformed with the recommended ideal thickness of a metal-ceramic restoration (1 - 1.5mm) (Yamamoto, 1985:25). Yamamoto (1985:297 -298) noted that the colour of the dentine layer was affected by the opaque colour, as well as by the amount of dentine applied. Basically the more dentine and incisal ceramic applied to the opaque layer, the greater the translucency of the specimen. Moreover, the reflectance of the underlying colour will have an effect on the overall shade of the restoration.

The circled area in Figure 4.9 shows the tolerance of 0.01 colour units. Samples falling outside this area were considered significantly different from both the gilded control group and the shade guide. Although the statistical analysis presented in Table 4.5 shows that all the P - values accepted that the sample groups and the gilded control group were similar, visual analysis confirmed that the sample groups and the gilded control group were indeed different. The difference was most noticeable in terms of hue. The gilded control group reflected a warmer (yellowier) hue than the sample groups when viewed. It is apparent that the results of the chromaticity co-ordinates and the human visual analysis consistently agreed, which would certainly imply that further investigation into the relationship between the chromaticity co-ordinates produced by the spectrophotometer and human visual analysis is required.

4. THE REFLECTANCE DIAGRAMS OF THE OPAQUE, DENTINE AND GLAZE FIRINGS

The reflectance graphs showed that from the opaque layering to the dentine and glaze firings the difference between the numerical values became increasingly smaller. Consequently, there was evidence to suggest that the more ceramic layers that were applied to the ceramic restoration the closer the hue, value and chroma values became among the different metal alloys. This was substantiated by visual viewing (Fig. 4.3 and 4.4). Jacobs *et al.* (1987:138) found that, when testing the colour of different amounts of applied ceramic on restorations, all detected differences were most obvious when thinner layers of dentine

were used; and as more ceramic was applied to the restorations' surface, it became increasingly difficult by means of spectrophotometer and human visual analysis to differentiate between two sample groups.

In summary the results showed that the colours of the base metal alloys were increasingly similar to one another after the application of the dentine layer. This suggests that after the application of the enamel and incisal layers, the differences in colour among the various base metal alloy restorations proved to be insignificant.

However it was found that, if the metal alloy foundation was of a different hue, like that of the gilded control group, then there were significant differences in the resultant colour.

- The purpose of glaze firing was to confirm the supposition that the oxide formation at the ceramo-metal interface was continuous and that it would therefore affect the final shade of the restoration. The results showed that the latter was true; however, the assumption that the change in colour was caused by the increase in oxide formation was invalid due to the findings:
- The light microscope results showed that there was no excessive build up of oxide at the ceramo-metal interface or excessive saturation of the oxide into the ceramic after firing.
- The penetration of the oxide into the opaque layer was normal for all base metal alloys, with the exception of Wirobond.
- The changes in value were proportional when comparing one sample group with the next. Therefore, if oxidation of the metal had been an influencing factor, the changes in value would have been variable.
- The control group showed a change in value which was proportional to the rest of the sample groups.

Since there was no excessive saturation of the oxide into the opaque, it is suggested that the change in value was due to changes in the porcelain grain boundaries. Many researchers (Phillips, 1982:515) have found that there is a tendency for porcelain to devitrify if excessive firing temperatures are reached, or if excessive firings are made. Although all firing cycle temperatures in this study were according to manufacturers' recommendations, and the number of firings were kept to a minimum, the change in reflectance value from the dentine firing to the glaze firing was evident. The differences in Y% between the dentine and glaze firing were small, and it is suggested that any further firings would have produced larger

differences in Y% value. When comparing the glaze firing results with those of the dentine firing result, it was found that the greening of the porcelain was unavoidable and was part of the ceramic firing process. It is therefore suggested that the greening effect on the porcelain can be kept to a minimum by avoiding unnecessary firings of the restoration.

In the light of the above discussions, the second hypothesis was rejected, based on the following:

- The reflectance curves of the opaque, dentine and glaze firings showed insignificant differences among the five base metal alloy groups.
- The Y% values of the opaque, dentine and glaze firings showed insignificant differences among the base metal alloy groups.
- The chromaticity diagrams showed that the colour compositions of the five base metal alloy groups differed within one colour unit from one another, and they were therefore classed as being similar in chroma.

5.4 DISCUSSION LIGHT MICROSCOPE RESULTS

During the spectrophotometer testing, the reflectance curves of the oxide firings showed differences in colour among the various sample groups. The most significant difference occurred in the control group which produced high reflectance values and bright yellow hues, which was expected due to the gold foundation layer. What was noticeable however was the fact that after the application of the opaque layer, the differences in colour among the base metal groups was reduced dramatically. However, there were still differences among the sample groups in terms of reflectance value. Crispen *et al.* (1991:356) explains this phenomenon as the oxide layer being dissolved into the opaque layer and thereby affecting the shade.

Annexure A provides sectional views of the five base metal alloys (A-E) and the control group (X). The alphabetical labels correspond with the numbering system that was explained in chapter three.

When visually viewing the base metal groups at 200x magnification, it was concluded that there was very little penetration of the oxide layer into the opaque. The only exception was Wirobond (Annexure A [C]), which displayed a uniform oxide penetration which was significantly greater than that of the rest of the base metal alloys. It was concluded that cobalt had an effect on the amount of oxide penetration into the opaque layer. This conclusion was based on the fact that

Wirobond, which was the only cobalt-chrome metal (Table 5.1) tested in this study, produced significantly different results when compared with the nickel-chrome alloys. Although Wirobond produced an oxide which penetrated the opaque layer, the tristimulus values showed that the oxide did not penetrate sufficiently enough to affect the colour of the opaque layer. Strandman (1982:74) reported that the chrome-cobalt alloy he tested produced small amounts of oxide and that the layer of such an oxide was easily removed. What was not reported in Strandman's report, was the amount of oxide that was absorbed by the porcelain, or the colour changes that the oxides underwent during ceramic firings. It should be noted, however, that the latter was beyond the scope of his project.

During the light microscope evaluation of the samples it was noted that none of the other base metal alloy groups produced excessive oxides, therefore the question was posed as to why the chromaticity values (Fig. 4.9) of the base metals differed significantly from that of the control group. It is suggested that the gilded layer of the control group (Annexure A, [X]) had a highly reflective gold surface, which therefore produced a marked increase in hue and chroma of the opaque when compared with the base metal alloys. In retrospect, the fact that the highly reflective golden hue of the gilded foundation placed on the control group was visibly different from the hue of the base metal alloy specimens, suggests that the opaque tested in this study may indeed have been slightly transparent, resulting in the reflection of the gilded layer of the control group. The extent to which new opaqueing systems are conducive to the penetration of both reflected and diffused light, is most certainly a valid topic for future research. This high reflectivity of the gilded layer can be substantiated by the reflectivity curves of the oxide firings as presented in Figure 4.2.

5.5 SUBPROBLEM THREE

The hypothesis for subproblem three, viz. that metal-ceramic base metal alloys would produce an excessive oxide of undesirable colour which would dissolve into the porcelain during the firing process, with the result that colour matching to the shade guide would be difficult, was rejected

on the following bases:

- The critical assumptions were invalidated (Section 1.2.4 [5]).
- The oxide colours were masked out by the opaque layer.
- No individual base metal alloy sample group could be singled out as being closer to the control group or the shade guide than any other.
- The control group was the nearest to the shade guide.
- The light microscope results indicated no migration of the oxide layer in the opaque layer.

5.5.1. ASSUMPTIONS

In the formulation of the study, the assumption was made that the effects of the increase in the production of the base metal oxides upon further firings, notably the dentine and glaze firings, would influence the shade as a result of the oxide dissolving into the porcelain.

This assumption was thought to be valid because local ceramists believed that the lighter shades (Vita shade classification: A1, A2) were being affected by an element within the metal-ceramic system. This element was believed to be the intermediate oxide layer dissolving into the porcelain.

However, the results of the opaque tristimulus readings showed that the oxide layer was adequately masked out by the opaque layer, and this result consequently invalidated the above assumption.

This effective masking of the opaque layer will be discussed in greater detail in the section to follow (section 5.5.{2}).

5.5.2. THE MASKING EFFECT OF THE OPAQUE LAYER

After the spectrophotometer testing of the oxide firings of all the base metal alloy groups, it was noticed that there were significant differences among the oxide colours of the base metal alloys (Table 4.1, Fig. 4.2). These results suggested that these dark coloured oxides could indeed have had an effect on the opaque and subsequent dentine layers. However, after the opaque layering process which was a requirement listed in the methodology of subproblem two, it was found that the masking effects of the opaque layer proved to be adequate with only a 2% difference among all five base metal alloy reflectance ranges (Fig. 4.3).

The results of the opaque firings in section four showed insignificant differences among the colours of the five base metal alloy groups (Fig. 4.3). It is evident in the literature (McLean 1980; Phillips, 1982; Yamamoto, 1985) that the oxide formation of base metal alloys is continuous during the different ceramic firing cycles. Therefore it was assumed that the effects of the base metal oxides would increase upon further firings, notably the dentine and glaze firings. However after the dentine and glaze firings there was no significant evidence (Fig. 4.10 & Fig. 4.4 respectively) to show that the oxide layer had affected the hue, value or chroma of the base metal alloys.

Upon closer evaluation of the opaque layer it became evident that the reverse of the original hypothesis had occurred. Originally it was hypothesised that the dark coloured oxide would affect the opaque and thereby affect the overall shade of the restoration. The results however showed an increase in whiteness of the base metal alloys, with the Y% values of the base metal alloys being closer to the E (pure white value: $x=0.333$ and $y=0.333$) value on the chromaticity diagram (Fig. 4.9), than the control group and the shade guide. Based on this observation it is suggested that the higher percentage reflectance was caused by the highly reflective opaque placed on a non-transparent metal foundation. This was in contrast with the shade guide which allowed the dispersion of light through the ceramic shade guide which had no metal foundation.

As noted previously, the basic hue of the metal foundation (section 5.3 [4]) was the determining factor in the final colour of the ceramic restoration. Although the base metal alloy samples showed whiter reflectance values than the gilded control group and the shade guide, the hue was the same for all the base metal sample groups with the only exception being Wirobond. However, the visual evaluation of the light microscope results (Annexure A [D]) revealed that Wirobond had a visibly thicker oxide formation than the rest of the base metal alloys.

The gilded control group reflected hues which were yellower (natural dentine colour) than the base metal alloys (Fig. 4.9) and the fact that the yellower coloured foundation reflected through the opaque layer suggested that the opaque layer was indeed slightly permeable to incident and reflected light. A possible explanation of the light permeable opaque layer may be the integration of the porcelain grain boundaries, which may have been due to the following:

- repetitive firing of the opaque, through the normal firing cycles
- high firing temperatures.

5.5.3. MATCHING THE BASE METAL ALLOY SAMPLES TO THE SHADE GUIDE

Matching the base metal alloy sample groups to the shade guide demonstrated the interrelationship between hue, value and chroma. Based on the findings, it was concluded that the assumption that the shade guide and the base metal alloy groups would have the same colour was invalid. An explanation of this interrelationship and its effects is as follows:

After the spectrophotometer testing of the final layer of the base metal alloys, the shade guide was tested in a similar manner. Following this the results of the base metal alloys and the shade guide testing were plotted on a chromaticity diagram (Fig. 4.9) and the results were evaluated. The purpose of this evaluation during this investigation was to use the objective information to find a base metal alloy which was the closest match to the shade guide in terms of hue, value and chroma.

When reviewing the results, it became clear that no single base metal alloy could compare with the shade guide. It was found that, overall, the Y% values of the base metal alloys were all too close to white and were highly reflective in nature when compared with the shade guide (Fig. 4.9). What was also noted was that the differences between the shade guide and the base metal alloys were significant, yet there was 1mm of dentine ceramic coverage. Jacobs *et al.* (1987) noted that the thickness of ceramic becomes the basis for control of colour in metal-ceramics. It is suggested that any restoration with less than 1mm of ceramic coverage over the opaque layer could increase the light reflectance significantly, to the extent that the ceramist might have to produce substantial correction to the manufactured restoration in order to achieve a suitable shade match to the shade guide.

The contribution of hue and chroma to base metal alloy metal-ceramics also differed significantly from the shade guide (Fig. 4.9). Based on this the following is suggested: that the light reflecting off the opaque layer (which was already noted as being significantly whiter than the shade guide) influenced the hue and chroma of the overall shade of the base metal alloys and therefore produced a lighter shade when comparing the base metal alloy samples with the shade guide. The second influencing factor was the fact that the shade guide allowed light dispersion and refraction through the ceramic medium to a greater extent than the base metal alloy samples. This light dispersion and refraction enhanced the colour of the shade guide in terms of hue and chroma by

allowing the internal reflectance of the pigments in the ceramic to be reflected without any influence from an underlying opaque layer.

It is therefore suggested that, when comparing the base metal ceramic restorations with the shade guide, the reflective opaque layer of the base metal alloy metal-ceramic system could be lighter than the shade guide. It may therefore be necessary (details in section 6.2) to make a shade correction to the base metal alloy opaque layer in order to appropriately adjust the colour of the highly reflective opaque layer.

5.5.4. MATCHING THE CONTROL GROUP TO THE SHADE GUIDE

The shade guide and the control group were made of different materials and manufactured according to different methods. This posed an important question, viz. why they produced such similar results.

When comparing the control group with the shade guide, there was a remarkable similarity in terms of hue and chroma. Although the chroma of the shade guide tended towards red, the hue of the control group was much closer to the shade guide than to the base metal alloy sample groups. The reason that the control group reflected more red wavelengths of light than the shade guide was possibly due to the reflected colour of the gilded layer. What was also noticeable was that the control group had a very high Y% value (Fig. 4.10). This 1% increase in value was even greater than that of the base metal alloy groups, which was also noticed by Yamamoto (1985:293), who found that this high reflectivity was caused by the overall reflectivity of the gilding system.

It is suggested that the gilding of semi-precious metal alloys be done only in areas where warm reflection of the restoration is required. Some of these areas might include lighter shades which have a substantial amount of dentine reflectance around the cervical third or darker shades which require greater reflectance from the underlying foundation.

5.6 SUMMARY

In Chapter four (section 4.2) the objective results showed that the oxides of the five base metal alloy groups tested, were indeed different in colour (Fig. 4.2). This observation suggested that the final colour of the base metal alloy restorations could have been affected by the different coloured oxide layers. After the testing of the opaque layer (Table 4.3 & Fig. 4.3) the results

showed no significant difference in colour among the base metal alloy groups. The only significant difference noted was the colour of the gilded control group which had a reflectance value which was 4% higher than that of the base metal alloy performing closest to it, viz. Ceradium V. A similar trend was noted for the dentine layering.

Since the commercial dental technician has no objective means of analysing the performance of his restoration in terms of colour, the final analysis of this study involved a combination of objective and visual evaluation. Therefore the final finding of this study is as follows:

- The hypothesis that the intermediate oxide layer in base metal alloys would affect the shade of a final restoration was rejected. It must be noted however, that if there is any significant change in hue of the metal (oxide/ intermediate layer)(whether from surface conditioners or gilding pastes in any metal-ceramic system), then the final shade of the restoration could be affected.

CHAPTER SIX

CONCLUSIONS, RECOMMENDATIONS AND PRACTICAL SIGNIFICANCE

6.1 CONCLUSIONS

1. By using base metal alloys for metal-ceramic restorations the dark coloured oxide layer produced by base metals will not affect the shade of a final restoration. It must be noted that the basic hue of the metal foundation is the determining factor of the overall shade of the final restoration. The hue of any metal foundation should not be radically different from the shade guide or a custom made shade guide, if a comparable match is to be achieved.
2. The spray-on technique of applying opaques produced results that showed consistency in terms of colour in the base metal alloy groups. The colour values of the base metal alloys were however lower than those of the control group and the shade guide, both of which produced warmer colours.
3. When dentine was applied to the specimens, the non-precious groups all had similar hue, value and chroma, and there were no significant differences among the sample groups.
4. The gilded control group matched the shade guide better than the base metal alloy samples. Although the control group and the shade guide matched in terms of hue and chroma, the control group had a higher Y% reflectance value (i.e. was lighter) than the custom made shade guide.
5. The gilded control group, in terms of tristimulus values, matched the mean natural tooth colour (580nm) better than the base metal alloy ceramic samples.
6. All the sample groups showed a decrease in value when the specimens were subjected to a glaze firing. The conclusion was that the ceramic grain boundaries were affected, resulting in a greening effect on the final shade of the ceramic.

7. This study concluded that, by applying a warm coloured gilding material, the effects of a dark coloured oxide will be reduced, which in turn will produce a shade match which is closer in colour to the shade guide. Such a close match is not possible without the use of a gilding material.

6.2 RECOMMENDATIONS

1. Further research is required into the effects of firing an oxide before the application of ceramics, and the effects of the shade if the oxide firing is left out of the firing process. An investigation should also be conducted into the effects of different oxide formations found on the metal surfaces.
2. During the testing of the base metal alloy oxide colours, it was noted that the nickel-chrome alloys produced darker coloured oxides than the cobalt-chrome alloys. Further research is required with regard to the content of the earth elements found in nickel-chrome and cobalt-chrome and the effect that these elements have on the oxide colours of the base metal alloys.
3. Within the budget constraints of this study, only one layer of the gilding paste was applied to the specimens. Further research should be carried out to determine of the effects of a second layer of gold gilding material.
4. It was noted that various gilding materials had different masking properties. It is recommended that pre-mixed gilding pastes be used as opposed to self-mixing products.
5. The differences in the spray-on and brush-on techniques of applying opaque materials should be tested in terms of the effect the two techniques have on the final shade of ceramics.
6. It is recommended that further research be carried out on the effects chrome-cobalt oxides have on the final shades of metal ceramics. This includes the measuring of the depth of saturation of the oxide into the opaque material, and the possible reasons behind the saturation of the oxide into the ceramic as opposed to other base metal alloys.

7. In order to achieve better results with base metal alloy ceramics, it is recommended that research be carried out in the mixing of opaque colours to increase the hue and chroma of the foundation of the base metal, metal-ceramic system.
8. It is recommended that further research be carried out on the differences between precious, semi-precious and base metal alloy oxides and the effects that these oxides have on the shade of final restorations.
9. There have been speculations in the local commercial industry that the greening effect of overfired porcelain may be reduced by lowering the firing temperature (840°C) for the initial dentine firings, followed by a higher glaze firing of 940°C. The reasoning behind the success of this method may be attributed to the fact that the vital colour pigments are maintained in position until the last firing. However, this is pure speculation and may lead to a reputable research project in the future.

6.3 PRACTICAL SIGNIFICANCE

For the commercial dental technician this study has highlighted the following facts:

1. The colour of metal ceramic restorations is significantly affected by the preparation of the metal foundation. In terms of base metal alloys the colour of a restoration will not differ significantly from one base metal alloy to the next; however, if metal-ceramic alloys are to be changed within the industry, one must ensure that the basic hue of the newly acquired base metal alloy oxide is similar to the metal which falls within the experience of the laboratory owner.
2. The colour effects of dark base metal oxides are removed after the application of opaque. However, the shade of the restoration will be significantly whiter than that of a semi-precious metal alloy whose metal surface has been prepared with a metal conditioner.
3. Metal-ceramic shades, specifically the lighter shades, will reflect whiter colours than the shade guide. It is therefore recommended that corrections be made to the opaque colours in order to change the hue of the final restoration to achieve the desired shade match.

4. It is recommended that a gilding layer be applied to semi-precious metals if a reliable shade match is required when comparing a restoration with natural teeth. This will produce restorations which are warmer in colour than those produced by base metal alloy ceramic systems. However, care should be taken when dealing with light shades.
5. Multiple firings should be kept to a minimum to avoid changes in value of the ceramic.

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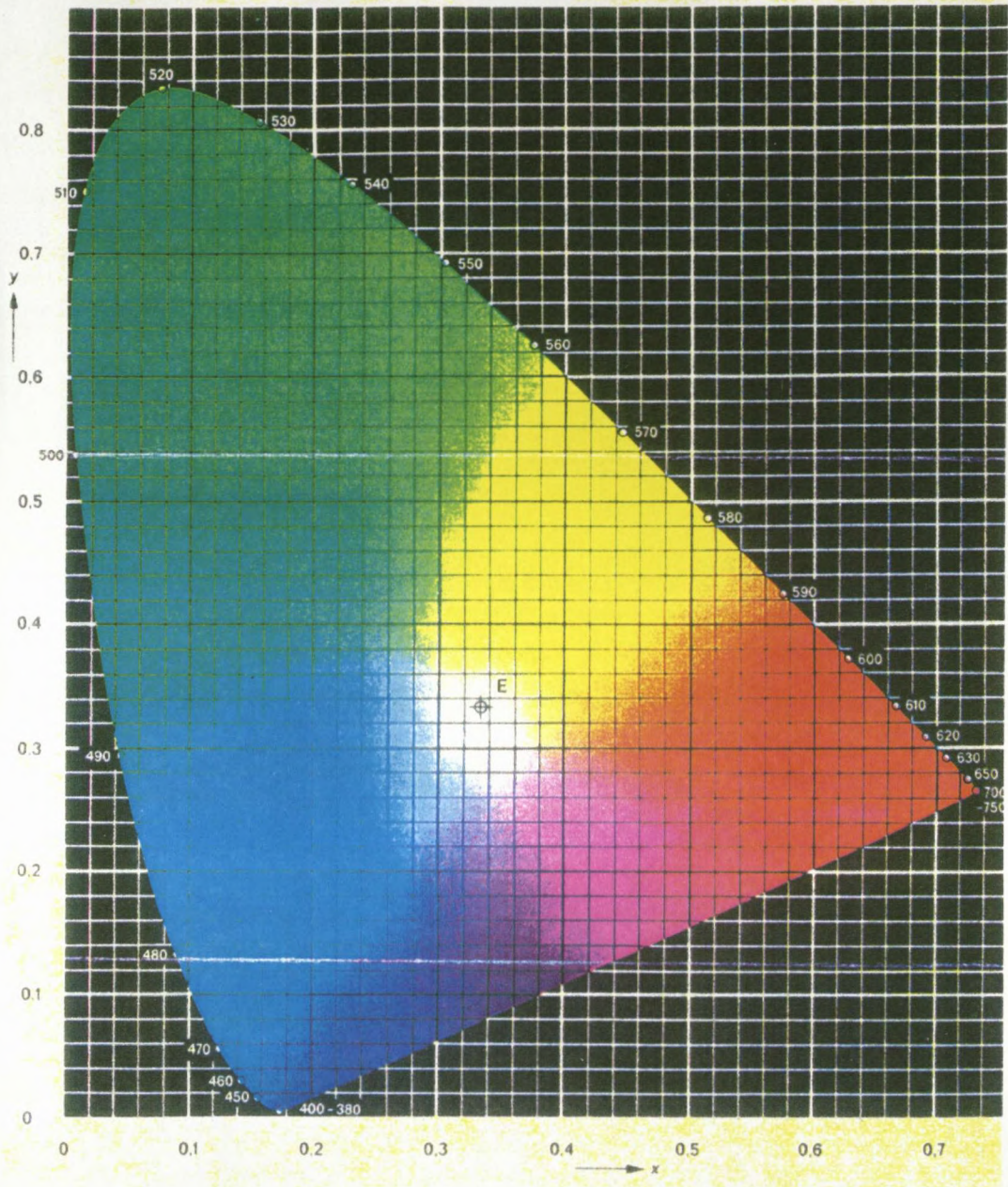
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ANNEXTURE A : Sectional Views of Specimens at 200x Magnification

- A = Ceradium V
- B = Wiron 99
- C = Wirobond
- D = Heranium
- E = Remainium CD
- X = Bond-on-4 (control group)

STATISTICAL VALUES OF THE TWO-SAMPLE ANALYSIS TEST				
METAL ALLOY	STD DEVIATION	COMPUTED t STATISTIC	VARIANCE	SIG.LEVEL
OPAQUE FIRING				
CERADIUM.	9.26918	0.641783	85.9178	0.52338
HERANIUM	9.09234	0.743498	82.6706	0.459989
REMANIUM.	8.85621	0.978349	78.4325	0.331704
WIROBOND	8.38872	1.46311	70.3707	0.148489
WIRON 99	9.26125	0.645535	85.7708	0.520962
DENTINE FIRING				
CERADIUM	9.88984	0.597217	97.8089	0.552538
HERANIUM	10.2746	0.406117	105.567	0.686055
REMAINUM	13.2131	1.10928	174.585	0.271595
WIROBOND	9.46455	1.08101	89.5777	0.283881
WIRON 99	10.2903	0.536879	105.891	0.593272
GLAZE FIRING				
CERADIUM	10.4394	0.348232	108.98	0.728846
HERANIUM	10.38	0.227047	107.744	0.821134
REMANIUM	10.1437	0.662422	102.895	0.510156
WIROBOND	9.4758	0.977035	89.7908	0.332349
WIRON 99	10.6444	0.389708	113.303	0.698088



ANNEXTURE C : CHROMATICITY DIAGRAM DIN 5033 (1964)