DEVELOPMENT OF *Cu-Al-Ni* ALLOY FOR DENTAL POST AND CORE APPLICATION

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DEVELOPMENT OF *Cu-Al-Ni* ALLOY FOR DENTAL POST AND CORE APPLICATION

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ABSTRACT

Generally, the technique used for replacement of the tooth is "posts and cores". Noble and semi-precious dental alloys have numerous excellent properties, but they are expensive and unaffordable for lots of patients. The copper-aluminum-nickel alloy is a reasonably priced alternative, and theoretically is perceived as corrosion resistant. Therefore, this situation could potentially be a prototype to develop a base-metal alloy for dental post and core application, and generate dentistry that is more affordable to patients.

Sixteen groups of experiment alloys, with varying proportions 0, 3, 6, 9 wt % Al and 0, 2, 4, 6 wt % Ni, were prepared and their properties were evaluated.

As generated by the biocompatibility characteristic, the 6 wt % Ni series was inappropriate for use for this purpose, due to its high toxicity. Also, the 12 wt % Al series was not suitable due to its high brittleness and low castability. Additionally, the 0 wt % Ni series revealed extremely low strength and elongation, and the 6 wt % Al series exhibited only a small amount of modulus to withstand mastication in the mouth. As a result, the 9 wt % Al with the combination of 2 wt % Ni and 4 wt % Ni series not only produced appropriate modulus of elasticity (63.9 ± 5.1 to 139.1 ± 14.2 GPa), predictable on the point of dental gold alloy, and presented higher corrosion resistance than the others, but also had a value of 0.2 % proof strength (180.4 ± 5.8 to 198.2 ± 3.4 MPa), which complied with the ISO standard of at least 180 MPa.

In conclusion, the 9 wt % Al in the combination of 2 wt % Ni and 4 wt % Ni series has the potential to present for dental post and core application, and is capable of being used for further development as good quality post-and-core material.

KEY WORDS: POST AND CORE / DENTAL ALLOY / COPPER ALLOY / BASE-METAL ALLOY / BASE METAL

142 pages

การพัฒนาโลหะเจือ ทองแดง อลูมิเนียม นิกเกิ้ล สำหรับเดือยฟันทางทันตกรรม DEVELOPMENT OF *Cu-Al-Ni* ALLOY FOR DENTAL POST AND CORE APPLICATION

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บทคัดย่อ

โดยทั่วไปแล้วโลหะที่ใช้ในการบูรณะพื้นสำหรับงาน post and core ทางทันตกรรม เป็น โลหะ เจือ ชนิด noble และ semi-precious ซึ่งมีกุณสมบัติที่ดีมาก แต่โลหะเหล่านั้นมีรากาแพง ผู้ป่วยจึงมีโอกาศ เข้าถึงการรักษาได้น้อย โลหะเงือทองแดง อลูมิเนียม นิกเกิล มีรากาที่ถูกกว่า และมีคุณสมบัติ ด้านทานการ กร่อนได้เป็นอย่างดี ด้วยเหตุนี้จึงเหมาะสมในการนำมาเป็นต้นแบบในการพัฒนาเป็นโลหะเงือชนิด basemetal เพื่อใช้ในงานชนิดนี้ซึ่งจะทำให้ผู้ป่วยได้มีโอกาศเข้าถึงการรักษามากขึ้น

โลหะเจือทองแดง อลูมิเนียม นิกเกิล จำนวน 16 กลุ่ม ถูกหลอมตามกระบวนการทางโลหะ วิทยา โดยมี สัดส่วนโดยน้ำหนัก ของ อลูมิเนียม 0, 3, 6, 9 % นิกเกิล 0, 2, 4, 6 % และทองแดงเป็นสัดส่วน สมดุล แล้วทำการทดสอบและประเมินคุณสมบัติของโลหะเจือทองแดงนี้ ตามที่ระบุใน ISO International Standard ที่เกี่ยวข้อง

ผลจากการทดสอบแล้วทำการประเมินพบว่าในกลุ่มที่มีสัดส่วนนิกเกิล 6 % มีความเป็นพิษสูง ที่สุด กลุ่มที่มีสัดส่วนอลูมิเนียม 12 % มี brittleness สูงมาก ในขณะที่มี castability ค่ำ เช่นเดียวกับกลุ่มของ อลูมิเนียม 6 % ทำให้ modulus ค่ำมากไม่สามารถด้านทานกับแรงภายในช่องปากได้ ส่วนกลุ่มที่ไม่มีสัดส่วน ของนิกเกิลอยู่เลยพบว่า tensile strength และ elongation ค่ำเกินไป ไม่เหมาะสมที่จะใช้สำหรับวัตถุประสงค์นี้

กลุ่มที่มีสัคส่วนอลูมิเนียม 9 % โคยมีนิกเกิล 2 % และ 4 % เป็นองค์ประกอบ มีค่า modulus อยู่ ระหว่าง 63.9±5.1 กับ 139.1±14.2 GPa เทียบเท่ากับสมบัติของโลหะเงือทอง และมีความต้านทานการกัด กร่อน สูงกว่ากลุ่มอื่นๆ อีกทั้งค่า 0.2 % proof strength มีค่าอยู่ระหว่าง 1**8**0.4±5.8 กับ 198.2±3.4 MPa สอดคล้องกับค่ามาตรฐานของ ISO ซึ่งกำหนดให้ต้องมีค่าอย่างน้อย 180 MPa

ดังนั้นโดยสรุป กลุ่มโลหะเจือทองแดง อลูมิเนียม 9 % ที่มี นิกเกิล สัดส่วน 2 % และ 4 % เป็น องค์ประกอบมีสมบัติเหมาะสมในการนำไปใช้บูรณะพื้น สำหรับงาน post and core ทางทันตกรรม และมี ศักยภาพในการที่จะนำไปพัฒนาเป็นวัสดุทันตกรรมที่กุณภาพต่อไป

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LIST OF ABBREVIATIONS

Δ a*, Da*	Chromaticness a* (redness) difference
Δ b*, Db*	Chromaticness b* (yellowness) difference
ΔE^* , DE*	Colour difference
Δ L*, DL*	Lightness difference
®	Registered
°C	Degree Celsius
7d	Seven days
А	Percentage elongation
Ag/AgCl	Silver/Silver chloride (Reference electrode)
Al	Aluminium
Al_2O_3	Aluminum oxide
Amps	Amperes
at.	Atomic
ATCC	American Type Culture Collection
CE	Counter electrode
CFRC	Carbon-fiber reinforced carbon
CIELAB a*	Chromaticness a* (redness)
CIELAB b*	Chromaticness b* (yellowness)
cm ²	Square millimeter
CO_2	Carbon dioxide
Co-Cr	Cobalt-Chrome alloy
Cu	Copper
DMEM	Dulbecco's Modified Eagle Medium
DNA	Deoxyribonucleic acid
Е	Electrical potential
Ec	Active peak potential

LIST OF ABBREVIATIONS (cont.)

EDTA	Ethylenediaminetetra-Acetic Acid di-sodium salt
E _{ocp}	Open circuit potential
Ep	Breakdown potential
et al.	and others, and elsewhere
FEPA	Federation of European Products of Abrasives
g	Gram
GMA	Glycid yl methacrylate
GPa	Giga Pascal
Ι	Electrical current
Ic	Active current density
in vitro	Made to occur in a laboratory vessel or other controlled
	experimental environment rather than within a living organism or
	natural setting.
Ip	Passive current density
ISO	International Organization for Standardization
Kgf	Kilogram force
L929	The American Type Culture Collection L929-3T3
Liq.	Liquid
μ	Micron
mbar	Millibar
min	Minute
ml	Milliliter
mm	Millimeter
μm	Micro meter
mol/l	Mole per liter
MPa	Mega Pascal
mV	Millivolts

LIST OF ABBREVIATIONS (cont.)

NaCl	Sodium chloride
NaOCl	Sodium hypo chloride
Ni	Nickel
Ni-Cr	Nickel-chrom alloy
NTG	N[p-tolyl] glycine
OCP	Open circuit potential
pН	pounds Hydrogenii
PMDM	Pyro mellitic di anhydride and 2-hydroxyethyl methacrylate
PMMA	Poly methyl meta acrylate
RE	Reference electrode
Rm	Ultimate tensile
Rp _{0.2}	0.2% proof strength of non-proportional extension
SCE	Saturated calomel electrode
SD	Standard Deviation
sec	Second
TM	Trademark
TMS	Thin metallic shafts
WE	Working electrode
wt %	Weight percentage

CHAPTER I INTRODUCTION

Endodontic treatment of missing tooth structure has been accomplished by using various methods for extended time. An approach commonly used by the dentists is to put the post and core onto the tooth. The post which was originally designed to retain the artificial crown was later shown as a system to provide resistance to fracture of the root.

The procedure frequently used to produce post and core may be fabricated from a dental casting alloy and generally are prepared as single units. They reproduce the contours of the prepared root canal and adapt well to the canal morphology and are commonly chosen in those situations where the canal is irregularly shaped.

A significant requirement for post and core materials is stiffness. The stiffness of the post (a combination of cross-sectional geometry and modulus of elasticity) is an exceptionally important characteristic. Insufficient stiffness of the post permits micro-movement of the core and distortion of the restoration at the margins during function. However, the post must not be so stiff that it cannot deform compatibly with the root as it bends under functional loads. Failure to do so, root fracture could be the significance.

The posts and cores, in clinical situation, are in the closed environment. They are encapsulated within the luting cement, root structure and the crown restorations. Some luting cement may form an insoluble film, cover the posts and cores and completely prevent them from contacting dentinal fluid, which may cause the corrosion of the post.

The developmental of aluminum-copper-nickel alloy will be functional in dental post and core application.

1.1 Statement of problems

Dental alloys are commonly chosen to fabricate the custom cast posts and cores. There are various types of alloys used in dentistry, high-precious, semi-precious and non-precious alloys. The advantages of high-precious and semi-precious dental alloys are good cast-ability and corrosion resistant, optimum hardness, strength, and elastic modulus that make them suitable for casting the custom cast post and core. The alloys are not too stiff and have yellow-gold color, especially the high-precious alloy.

However the alloys are expensive and not affordable by lots of patients. Because of the financial problem in some patients, the non-precious dental alloy somehow cannot be avoidable. The disadvantages of available non-precious dental alloys are their poor cast-ability due to its low density, very high hardness, stiffness, and elastic modulus. Therefore, the non-precious alloy post and core cannot deform compatibly with the root under functional loads and may cause the root fracture. The development of new non-precious dental alloy that is more favorable for post and core application will not only make dentistry more affordable to the patients, but also give dentists more choices of material for quality work.

Therefore, the effectiveness of the experimental aluminum-copper-nickel alloy will be limited only for dental post and core application at the beginning.

1.2 Objectives of the study

1.2.1 Part 1

- 1. To develop the copper aluminum nickel alloy for dental post-and-core application.
- 2. To evaluate thermal properties of the experimental alloy.

1.2.2 Part 2

- 1. To evaluate surface hardness of the experimental alloy.
- To evaluate Tensile strength, Elastic of modulus, Elongation and 0.2% Proof strength of the experimental alloy.

1.2.3 Part 3

- 1. To evaluate corrosion resistance and electrochemical behavior (Potentiodynamic polarization) of the experimental alloy.
- 2. To evaluate tarnish resistance (Static and Dynamic immersion) of the experimental alloy.

1.2.4 Part 4

To evaluate bio-compatibility of the experimental alloy.

1.3 Clinical implications

The developed aluminum-copper-nickel alloy will be accessible well with dental post-and-core application. Additionally, providing practical price and also affordable by lots of patients.

1.4 Hypothesis

The developed alloy has a high potential to be an alloy for dental post and core application in term of:

- 1.4.1 Presenting appropriate mechanical properties, consistent with the ISO 22674:2006.
- 1.4.2 Inducing proper corrosion resistance and simulating satisfactory corrosion behavior in relation to the ISO 10271:2001.
- 1.4.3 Providing acceptable toxicity: The value of cell response not in excess of the positive control value in accordance with the ISO 10993-5:1999 and 7405:2008.

CHAPTER II LITERATURE REVIEW

Endodontic treatment has provided dentistry with the ability to retain teeth that only a few decades ago would have been extracted without hesitation. The restorative method for endodontically treated teeth depends on the amount of supported coronal tooth structure which remains. If substantial coronal tooth structure is intact, a conservative approach may be used. If little or no coronal tooth structure remains, provision must be made for developing a core reconstruction over which a crown can be placed.^{1, 3, 12}The replacement of missing tooth structure has been accomplished by using various methods for thousands of years. The Franks (200-737 A.D.) described the use of a wooden dowel placed in the root to provide an anchor for the artificial crown.¹³ Over 250 years ago, Pierre Fauchard described techniques of "pivoting teeth" to allow a precious metal post to be fitted and secured into a canal which was prepared with a watchmaker's reamer.¹⁴

2.1 Anatomical and biological considerations

Endodontically treated teeth have special needs that exceed the requirements of teeth with vital pulps.¹⁵ These needs are related to the role of moisture loss and the nature of dentin, alterations in strength caused by architectural changes in the morphology of the teeth, concepts of biomechanical behavior of tooth structure under stress, the nature of dentin toughness in pulpless teeth, and changes in collagen alignment. Helfer et al.¹⁶ reported that there was 9% less moisture in the calcified tissues of pulpless teeth compared to vital teeth. Greater loss of moisture was found in anterior teeth than in posterior teeth. This study was the first investigation lending credence to the empirical assumption that pulpless teeth may have increased brittleness from moisture loss. In 1992, Huang et al.¹⁷ demonstrated that the dehydration of dentin increased the Young's modulus, proportional limit (in compression), and especially

the ultimate strength (in both compression and tension). Substantial dehydration changed the fracture characteristics of dentin specimens under static compressive and indirect tensile loading. Fifty percent of the dentin specimens from endodontically treated teeth exhibited greater plastic deformation than normal dentin in compression. These results did not support the theory that dehydration after endodontic treatment per se weakens the dentin in terms of reduction in compressive and tensile strengths.¹⁷

Reeh et al.¹⁸ evaluated the effects of endodontic treatment and conventional restorative procedures on tooth stiffness. Endodontic treatment reduced tooth stiffness by only 5%, and this reduction was contributed entirely to the access opening. Restorative procedures resulted in significant loss of tooth stiffness, and cavity preparation that disrupted the continuity of the marginal ridge caused the greatest loss of strength. Carter et al.¹⁹, in 1983, evaluated shear strength and toughness values from dentin of vital and endodontically treated teeth. The shear strength and toughness values form pulpless teeth were lower and significantly different from the values obtained for dentin of teeth with vital pulps. There was approximately 14% reduction in the strength and toughness suggested weakness and brittleness of pulpless teeth.

The loss of both internal and external tooth structure, from caries excavation, access cavity preparation, and removal of radicular dentin during cleaning and shaping or post space preparation weakens the endodontically treated tooth considerably.²⁰ Gutmann^{15, 21} has demonstrated that the biomechanical behavior of a tooth changes after access-cavity preparation because of the loss of the prestressed state in its laminated structure. The tooth can deform to a greater extent under applied loads, and it becomes more susceptible to fracture.

Recently, many reports have been published with descriptions of materials and methods intended to assist the dentist in the restoration of the pulpless tooth.^{4, 13, 22, 28} There are four components that may be applied when restoring endodontically treated teeth: posts, pins, cores and the final restorations. All of these play a role in restoring the teeth to form and function.¹³

2.2 Posts or dowels

Endodontic posts or dowels are usually cemented or threaded into a prepared root canal. The main function of a post placed in the root canal of the endodontically treated tooth is to provide retention for the core that is substituting for coronal tooth structure and supporting or retaining the final restoration.^{13,22-24,29} The post will provide retention to the core by distributing the forces of occlusion through the center of the root.

There are two broad classifications into which endodontic posts may be divided: custom posts and prefabricated posts. Techniques available for constructing a post and core are: 1) the custom or morphologic cast post and core that is cast as a single unit so the shape of the post conforms to the morphology of the prepared root canal^{13,22}; 2) a prefabricated commercial post which is available in various designs and is commonly used with various direct core materials^{13,22,24}; and 3) a cast post and core in which the post is cast from a premade burnout pattern and is identical to a prefabricated commercial post.^{13,23}

Custom cast posts and cores may be fabricated from a variety of materials and generally are prepared as single units. They reproduce the contours of the prepared canal and adapt well to the canal morphology and are commonly chosen in those situations where the canal is irregularly shaped.^{13,22,30,31} Favorable internal adaptation of the post will distribute the internal stresses relatively uniformly without stress concentration.³ The cast post and core will often provide greater resistance to rotation than a prefabricated post because of its asymmetrical shape, the incorporation of keyways or pins, and the extension of the casting over tooth structure. This resistance to rotation results in greater stability of the cast post and core.

Sorensen and Martinoff^{12, 32} suggested that the failure rate of teeth with cast posts and cores was greater than that of teeth crowned without post reinforcement. However, a closer evaluation of the data in their study by Morgano and Milot³¹ indicated that almost half of the cast posts were half the desired length or less, and their reported data strongly suggest that failure was the result of the compromised length of the cast posts in the study and not a condemnation of the cast post and core technique independently. Other recent in vivo studies also reported lower failure rates for cast posts-and-cores, and supported the benefit of custom cast posts-and-cores.³³⁻³⁵

Another method that can be used to fabricate a custom cast post and core is the "cast-to technique", whereby a metal core is cast to a stainless steel post. Sorensen et al.³⁶, in 1990, investigated the effect of heat treatment during the burnout procedure on the corrosion of the stainless steel post. They reported that prefabricated stainless steel posts subjected to simulated burnout procedures resulted in a noticeable reduction in corrosion resistance, and suggested that direct casting to stainless steel posts was contraindicated.³⁶

The use of a prefabricated dowel with a cast or direct core build-up offers the following advantages:

• A wrought or drawn dowel has superior strength compared with a cast core, especially when the dowel is less than 1.5 mm. in diameter.³⁷

• The use of these prefabricated dowels in combination with matching twist drills ensures a precise fit and, therefore, better retention than what is commonly achieved with tapered cast posts.²⁹

Conventional custom-cast posts-and-cores result in higher costs because they are made in a dental laboratory and additional chair time is required. Recently, the use of prefabricated posts has become more popular, and great variety of prefabricated posts has been developed. Most variations in design are attempts to satisfy the need for both retention and protection. Prefabricated posts can be parallelsided or tapered, smooth or serrated, cement type or threaded type, or any combination of these. Serrated surfaces provide mechanical undercuts for cement and significantly increase retention of parallel dowels over the retention obtained from smooth surfaces.⁶ Standlee et al.⁶ reported that pretapped, parallel-sided, threaded dowels were twice as retentive as a parallel-sided serrated dowels and approximately 6.6 times as retentive as smooth-sided tapered dowels. The presence of threads on posts greatly enhanced the retention of the cemented post over both smooth-sided and serrated posts,^{6, 38} but the use of prefabricated threaded endodontic posts is controversial. Other studies have shown that high stresses occurred during the tapping procedure and cementation, and frequent cracking of specimens resulted from these stresses even under ideal laboratory conditions.^{12, 39}

Cohen et al.⁴⁰, in 1992, investigated the effects of cold treatment on the physical properties of stainless steel and titanium-alloy endodontic posts and

instruments. Their results indicated that there was an increase in strength of stainless steel posts, reamers and external wrenches as a result of the single and double cold treatments at -96 $^{\rm O}$ C. Titanium posts did not result in changes in strength following a single cold treatment, but they were weakened by a double cold treatment.⁴⁰

King and Setchell⁴¹, in 1990, evaluated a prototype carbon-fiber reinforced carbon (CFRC) prefabricated post for the restoration of pulpless teeth with artificial crowns. Their results indicated that prefabricated CFRC posts exhibited properties comparable to, and in some cases better than, those of existing prefabricated posts. The mode of failure of specimens restored with CFRC posts was more favorable to the remaining tooth tissue compared to specimens restored with a metallic post.⁴¹

Teeth are subjected to different forces depending on their positions in the mouth. Maxillary anterior teeth commonly require posts to withstand shear stresses. Posterior teeth must primarily resist compressive stresses. Parallel-sided, serrated, cemented dowels have been reported most capable of distributing stresses to the radicular dentin when subjected to axial or inclined loading.⁷ This dowel design was seen to distribute loads better than other dowel systems.⁴² Yaman and Thorsteinsson⁴³, in 1992, demonstrated that cylindrical posts were intruded and created high apical stresses with vertical and inclined loading. Stiffer core materials diminished this intrusion but increased the cervical stresses.⁴³

In 1978, Standlee et al.⁶ studied the effects of cement, dowel length, diameter, and design on the retention of endodontic dowels. They consolidated concepts of dowel retention by using some current prefabricated post systems incorporating four retentive factors that are normally within the control of the dentist. Among the three dowel designs (tapered dowel, serrated cemented dowel and threaded tapped dowel) the tapered dowel was least retentive. The threaded tapped dowels showed the greatest retentive ability. They also reported that the effect of embedment depth of the dowels on retentive capacity was significant. In most instances, the more deeply the dowels were placed into their dentin channels, the more retentive they became. The effect of cement type was statistically insignificant except with the tapered type of post. Zinc phosphate cement was the most retentive, carboxylate cement was in an intermediate range, and the epoxy cement was the least retentive. From their study, the results indicated that the main factors influencing prefabricated

dowel retention are dowel design and depth of embedment. The type of cement and dowel diameter seemed to have little effect.

2.3 Pins

Pins can be used either alone or in combination with a post to provide retention for core material. The use of pins in situations where a post cannot be placed may provide additional retention for the core reconstruction. Pins can also be used in combination with a post to augment retention where a tooth has little remaining coronal structure.^{44,45} This application usually occurs in molars where a post might be placed in the distal or palatal canal with pins on the mesial or buccal aspect.¹³

A pin can improve resistance to dislodgment of the core as a result of the forces of mastication. Pins do not "reinforce" silver amalgam or composite resin restorations. Observations of clinical failures of silver amalgam restorations coupled with structural research studies, have led many investigators to the conclusion that the use of pins does not strengthen silver amalgam. In most cases restorative materials are weaker with pins than without pins.^{46, 47} Kao48 investigated the influence of pins on the fracture resistance of core materials, and the results indicated that incorporation of pins weakened both silver amalgam and composite resin. The reduction in strength was greater for silver amalgam than for composite resin.

Pins are placed within dentin most often in vital teeth, and therefore must be sufficiently small so as to encroach neither on the pulp nor on the external tooth surface. Three types of pins are available: self-threading, friction-lock, and cemented. The cemented pin systems employ serrated, metal rods that are cemented in oversized channels. Various pin-channel combinations are available. The friction-lock system utilizes a pin that is forced into an undersized channel. The self-threading pin systems consist of pins that are threaded into slightly undersized channels.⁴⁴ The self-threading pins are the most retentive. Pins which are forced to bind in their channels and create a friction lock are intermediate retainers. The cemented types are the least retentive.⁴⁹

2.4 Cores

The core replaces coronal tooth structure that has been lost because of caries and/or previous restorations. It provides a base that has sufficient bulk and retention to support the final restoration. The core can be formed from various materials or may be cast in precious or non-precious alloys in combination with the post.¹³

Before the introduction of prefabricated posts, the only method available was to cast a post-and-core in metal by the direct or indirect method. The post preparation was slightly tapered to provide ready withdrawal of the impression or direct pattern and unrestricted insertion of the casting.²³ A custom cast post and core can be made by coating a plastic sprue with acrylic resin, or a metal pin with wax, to form the post pattern. The pattern can be direct, fabricated on the tooth intraorally, or it can be indirect, fabricated on a cast made from an impression of the prepared canal.³⁷ This casting has superior physical characteristics at the junction of the core and the post. When much tooth structure is missing, the core may be required to provide an anti-rotational function to the restoration. The customized cast post and core fills the prepared root canal space and is designed to resist torsional forces.³¹ The cast post-and-core combination can utilize unconventional preparations in the dentin or auxiliary retentive pins or a cervical collar at the base of the core for anti-rotational purposes.³⁰ Casting a core to a stainless steel preformed post is not recommended.³⁶

A core restoration is often required to achieve satisfactory resistance and retentive form for a cast restoration.⁵⁰ Because the core becomes an integral part of the load-bearing structure of the tooth, it should provide sufficient strength to resist intraoral tensile and compressive forces. It should also be biocompatible, as well as chemically and dimensionally stable. Favorable marginal adaptation and ease of manipulation are other important demands. Silver amalgam, composite resin and reinforced glass ionomer restorative materials have been suggested as core materials to be placed in conjunction with prefabricated posts. Christensen⁵¹ stated, in 1993, that custom cast posts and cores were quite acceptable with proven usefulness. However, he claimed that prefabricated posts and cores rapidly, because prefabricated posts with

direct cores were faster and easier to construct but still provided acceptable strength and serviceability at a relatively low cost to the patient.⁵¹

Molars can often be restored with a corono-radicular silver amalgam core followed by a complete veneer crown or partial coverage cast preparation.⁵² With this method, silver amalgam is condensed 2 to 4 mm. into each canal and into the pulpal chamber and any remaining coronal portion of the tooth. Prefabricated posts are usually indicated to augment the retention of the silver amalgam core when the pulpal chamber is shallow.^{13, 23} Conclusions reported by Lovdahl and Nicholls⁵³, in 1977, indicated that pin-retained silver amalgam cores in molars required more force to dislodge than cast posts and cores. Gordon and Metzger⁵⁴ compared four designs of silver amalgam-core anchorage for their shear strengths. In their study, TMS-pin-retained cores (Coltene/Whaledent Inc. Mahwah, NJ) resisted higher shear forces than post-retained silver amalgam cores. Kern et al.⁵⁵ reported that a silver amalgam core with cemented posts was stronger than a core reconstruction in which silver amalgam only was used to create a dowel and core.

The mechanical properties of silver amalgam when used as core material are satisfactory. However, its relatively high coefficient of thermal expansion, the need for matrix bands during condensation and the inability to complete crown preparation in the same session are disadvantages.⁵⁶ The major factors limiting the longevity of silver amalgam restorations are operator- and patient-related. Both factors will also influence the longevity of any restoration.⁵⁷

Composite resins were introduced in the early 1960's. They are favored by many dentists because of their ease of manipulation, their ability to bond to tooth structure and their rapid setting time.⁵⁸ Composite resins are provided both as autocuring and light-curing materials.⁵⁶ In addition to the curing method, there are also microfilled products, macrofilled products and the newer hybrids. The hybrids are recommended as core materials. Their disadvantages include their higher coefficient of thermal expansion relative to that of enamel and the possible contamination by eugenol-containing temporary cements.⁵⁶ Micro-leakage and dimensional instability are also major disadvantages that may affect the longevity of the final cast restoration.^{58,59} Composite resins may be injected into the canal and onto the coronal tooth structure to form a one-piece post and core.^{13, 23} Many believe that this method will result in a post and core that lacks sufficient rigidity during function to prevent marginal leakage with the final restoration. Composite resin and silver amalgam posts are usually placed into post channels of limited depth to facilitate removal by the dentist if re-treatment of the root canal becomes necessary. However, the retention is reduced because of the shallow embedment depth, although some dentists have advocated placing retentive areas within the prepared post channel.^{13, 23}

Steele²⁸ claimed that the advantages of a reinforced composite resin post and core were: 1) its fabrication is less involved than that of the cast post and core; 2) its cost is minimal when compared to that for making gold castings; 3) it reduces the possibility of perforation of the root during pattern construction, because undercuts within the canal become advantageous; 4) it negates the hydraulic back pressure that can occur when cementing smooth-sided parallel posts⁶⁰; and 5) it negates the potential wedging effect of tapered posts and the unfavorable stresses associated with threaded posts. None of these purported advantages were based on controlled studies and were merely the opinion of the author.

Chang and Millstein⁶¹, in 1993, reported that composite resin with a posthead covering of 3 mm. was the most resistant to compression, whereas silver amalgam was the most resistant to tensile forces. The addition of titanium to composite resin as a reinforcement filler did not improve resistance to either compressive or tensile forces.⁶¹ However, a recent study reported by Cohen et al.⁶² investigated the fractural load of four core materials supported by five post designs. Their results indicated that for all posts, Tytin silver amalgam and Ti-Core material were significantly stronger than Ketac-Silver and G-C Miracle Mix material, and also suggested that Ti-Core composite material is at least as strong as Tytin silver amalgam.⁶² Nevertheless, the samples were loaded directly on the cores without a casting so the clinical relevance of this study is questionable.

Although the use of composite resin for cores has been successful, microleakage at the interface between the composite resin and the dentin root face can pose a serious problem.⁶³ If the interval of time is brief between making the core and cementation of the final coronal superstructure, micro-leakage can be minimized, provided that the temporary restoration affords an acceptable seal.²³ During 1970's and 1980's, research indicated that micro-leakage occurs at the interface between dentin and composite resin and cannot be completely eliminated by commercial dentin bonding systems, whereas the interface between dentin and glass ionomer cement displays no microleakage.⁶³⁻⁶⁵ Glass ionomer cement forms a chemical bond with dentin and leaches fluoride ions. Hence glass ionomer cement appears to be the material of choice to make leakproof cores.²³ The "sandwich" technique has been recommended for the core whereby Miracle Mix glass ionomer is placed between the dentin-root face and the composite resin after installation of the post.⁶⁶ The use of the Miracle Mix glass ionomer cement and composite resin provides a core with a seal at the core-dentin interface and the strength necessary for the preparation of the core.

Newer-generation composite resin materials have been developed, and most properties have been improved. Recently, composite resins are available to provide cariostatic restorations that are ultraconservative.⁶⁷

Glass ionomer cements were introduced in 1972.⁶⁸ The prime advantages of glass ionomer application are their fluoride release, adhesion to both enamel and dentin, and a coefficient of thermal expansion that is similar to that of dentin.^{56,69-72} However, their low wear resistance, low tensile strength and brittleness precluded their use as supportive core materials.^{59,73} Wilson and Kent^{68,74} have shown that sintered cermet (ceramic/metallic) composition could improve the mechanical properties of the glass ionomer, especially by increasing ductility and energy to fracture. The compressive strength, abrasion resistance, and flexural strength have also been found to be significantly increased with the addition of silver alloy powder.⁷⁵⁻⁷⁷ However, other studies suggested that these products are not strong enough to be used as core materials.^{59, 78, 79} Kovarik et al.⁵⁹, in 1992, examined the fatigue life of three core materials with cemented cast crowns under simulated chewing conditions. Silver amalgam cores had the lowest failure rate, followed by composite resin cores. All teeth restored with crowns over glass ionomer cores failed in the study.⁵⁹

Cohen et al.⁷³, in 1992, examined the shear bond strength to dentin of Ti-Core titanium reinforced composite resin and two third-generation dentinal bonding agents. Tenure dentinal bonding agent with Ti-Core composite resin had consistently greater shear bond strength than Scotchbond 2 dentinal bonding agent with Ti-Core composite resin. The shear bond strength for Tenure dentinal bonding agent with Ti-Core composite resin was two and one-half to three times greater than the strength of the silver-filled glass ionomer cements, Ketac-Silver glass ionomer cement and GC Miracle Mix glass ionomer cement.⁷³

In 1991, Millstein et al.⁸⁰ studied the retention between a serrated steel post and different core materials. Composite resin and silver amalgam were strong in tension and relatively fracture resistant whereas glass ionomer cement core materials were weak in tension with poor fracture resistance.⁸⁰

Brandal et al.⁸¹ compared the failure loads of endodontically treated anterior teeth restored with pin-retained silver amalgam restorations, Para-Post dowels and composite resin cores, and glass ionomer/silver amalgam alloy coronal-radicular cores. From their studies, the Para-Post dowel and composite resin core exhibited the highest mean failure load, and glass ionomer/silver amalgam alloy coronal-radicular cores recorded the lowest mean failure load. The investigators suggested that the restoration of endodontically treated anterior teeth with cores made from glass ionomer/silver amalgam alloy is contraindicated.

2.5 Final restoration and ferrule effect

The final restoration restores external tooth contour and allows harmonious function with those teeth remaining in the oral cavity. The design of the final restoration and the material from which it is fabricated vary considerably.¹³ A ferrule or encircling band of cast metal around the circumference of the tooth has been suggested to improve the integrity of the endodontically treated tooth. Barkhordar et al.⁸², in 1989, examined the effect of a metal collar with approximate 3 degrees of taper on the resistance of endodontically treated roots to fracture. Their findings indicated that reinforcement with a metal collar is necessary to enhance resistance to root fracture.

Various ferrules have been promoted, such as two-plane preparation of the root surface and a contrabevel around the occlusal surface of the preparation of a pulpless tooth.^{37, 83, 84} The purpose of the ferrule is to improve the structural integrity of the pulpless tooth by counteracting the functional lever forces, the wedging effect of

dowels and the lateral forces exerted during insertion of the dowel.⁸⁵ One millimeter of coronal tooth structure above the crown margin substantially increased the fracture resistance of pulpless teeth, whereas a one-mm contrabevel at either the tooth-core junction or the crown margin was ineffective.⁸⁶

In 1995, Libman and Nicholls⁸⁷ examined the load fatigue of teeth restored with cast posts and cores and complete crowns. Their results indicated that ferrule lengths less than 1.5 mm. to 2.0 mm. were ineffective in protecting the marginal integrity of the cast crowns. Loney and coworkers⁸⁸, in 1990, used three-dimensional photoelastic analysis to evaluate the effect of the ferrule design on stress with cast posts and cores. Their results indicated that, on a point-by-point basis, stresses were higher in the collared specimens, but variation in stress magnitude among five preselected points was greater within the non-collared group. In a study by Kern et al.⁵⁵, the addition of crowns to the cores did not produce a significant increase in the shear strength of their specimens.

Assif et al.²⁷ examined the effect of post design on the fracture resistance of endodontically treated premolars restored with cast crowns in vitro. They suggested that post design did not influence the fracture resistance of endodontically treated teeth, provided the core was covered by a complete cast crown with a 2 mm. margin or ferrule on healthy tooth structure. The crown changed the distribution of forces to the root and the post-and-core complex, rendering the post characteristics insignificance. In their study, the tooth supporting system with artificial periodontal membrane was not simulated. This experimental design may have had an influence on the fracture resistance of the teeth.

Hemmings et al.³⁰ investigated the resistance to torsional forces of various post-and-core designs. They reported that the cervical collar was the most favorable design, embracing resistance and reducing tooth fractures. Hunter and Hunter⁸⁹ suggested that it is unnecessary to incorporate a ferrule effect as part of a post-and-core foundation itself. The height of the remaining tooth structure between the margin of the core and the crown margin is the significant factor in determining the fracture resistance of endodontically treated teeth. The combination of a post-and-core restoration with an overcasting to act as a ferrule is commonly accepted today as a rational approach to corono-radicular stabilization for a pulpless tooth.^{85,90}

2.6 Concepts of reinforcement and tooth

A study reported by Kantor and Pines¹ indicated that an intraradicular post doubled the fracture resistance of the tooth. In 1979, Guzy and Nicholls⁷ compared the breaking loads of endodontically treated teeth, with and without cemented posts, to determine if the post reinforced the root against fracture. Their study reported that a statistically significant reinforcement had not been demonstrated by cementing a Kerr Endo-Post No. 100 into a sound endodontically treated tooth. Their analysis of post placement from an engineering viewpoint described how post placement at the center of the tooth could not reinforce the tooth under externally applied loading. Trabert et al.⁵, in 1978, reported that there were no significant differences in resistance to root fracture between untreated and endodontically treated teeth. They also concluded that preservation of internal tooth structure and the use of narrower posts provided maximal resistance to fracture. The amount of tooth structure that remains after endodontic and post preparation appears to be of prime importance.^{5, 12}

The restoration for the pulpless tooth should increase the resistance to horizontal and vertical forces.⁹¹ Coverage of the entire occlusal surface of the tooth with a restoration can reduce the incidence of vertical fractures.⁹² Lovdahl and Nicholls⁵³, in 1977, compared the strengths of two coronal restorative procedures with instrumented but unrestored teeth. They determined that endodontically treated teeth with unrestored, intact natural crowns were more resistant to fracture than teeth with pin amalgam cores and cast gold posts and cores. Sorensen and Martinoff³² conducted a retrospective study of 1273 endodontically treated teeth and concluded that there was no significant increase in resistance to fracture or dislodgment gained with intracoronal reinforcement, and coronal coverage did not significantly improve the rate of clinical success for maxillary and mandibular anterior teeth. However, the prognosis of pulpless maxillary and mandibular premolars and molars was significantly increased with coronal coverage. Currently it is advisable to place coronal coverage on endodontically treated posterior teeth. A restoration that covers the cusps, such as an onlay, is the most conservative means of restoring the structural integrity of the pulpless tooth.⁸⁵

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McDonald et al.⁹³ tested the impact fracture resistance of root-treated mandibular incisors restored with either stainless steel or experimental carbon fiber reinforced carbon (CFRC) prefabricated posts. Their results suggested that there was no advantage relative to fracture mechanics in restoring intact root-treated teeth with either stainless steel or CFRC rods. A three-dimensional finite element analysis by Ho et al.⁹⁴ in 1994 evaluate the effects of posts on stress distribution in dentin. Their study demonstrated that the stress distributions in dentin were similar whether or not the post was present, and the reinforcement effects from posts appeared limited in pulpless incisors. Trope et al.²⁰ in 1985 reported that the preparation of a post space significantly weakened endodontically treated teeth and that posts did not significantly strengthen them. However, filling the post spaces and the access cavities with a composite resin after acid etching of the root canals and cavity walls ensured strengthen the teeth.

A common aspect of most reinforcement studies was their destructive nature. Whether the tooth was actually reinforced was not studied. Dentin behaves according to Hooke's law.^{95, 96} When teeth are cyclically loaded within their proportional limit, there is complete elastic recovery, and they show an actual increase in strength and rigidity.⁹⁷ The effect of various post lengths on the strength or rigidity of the root within the elastic limit of dentin was evaluated by Leary et al.⁸ in 1987. They demonstrated that the increase in rigidity from the non-posted to the posted condition for their test samples was not statistically significant. Post-restored teeth were evaluated to determine the reinforcement capabilities of various post lengths. Their results suggested that: 1) as internal tooth structure was removed from the tooth, the tooth became weaker; 2) teeth with posts showed more reinforcement than non-posted teeth with the same manipulation; and 3) some load transfer appeared to exist with cemented posts.⁸

Felton et al.²⁵ investigated and compared the potential for root fracture resulting from the cementation of nine threaded and three non-threaded endodontic dowel systems. Their results indicated no statistically significant differences between dowel types when compared with each other, regardless of dowel shape, taper, or presence or absence of threads, or when compared to instrumented, non-obturated controls. They also suggested that the amount of remaining dentin and existing root

morphology may be a determining factor for endodontically treated teeth to resist fracture during dowel placement.

Sorensen and Engelman⁹⁸ investigated the effect of post adaptation on fracture resistance of endodontically treated teeth. Their results indicated that maximal adaptation of the post to the residual root structure with an overly tapered post preparation significantly increased the failure threshold of endodontically treated teeth, but upon failure the teeth were nonrestorable. Tapered posts resulted in fractures that were directed more apically and lingually. Parallel-sided posts had a lower frequency of fracture upon failure, involving less tooth structure. Parallel-sided posts surrounded by large amounts of cement had no significant effect on failure loads. The teeth in this study were prepared with over-tapered funnel-shaped canals, a situation that is sometimes encountered but relatively uncommon.

One of the possible causes of fracture of the root is excessive seating pressure exerted on the post and core during cementation. An accepted technique for cementation is: 1) depositing the rather liquid mixed cementing medium on the canal wall with a lentulo spiral; 2) coating the post with a thin film of the cement; and 3) slowly seating the post in the preparation. The last step must not compress the cementing medium forcefully; otherwise, hydraulic pressure may cause a fracture of the root. The post should be slowly teased into place, allowing the post to gently rebound so that the excess cement cans escape.²³

Factors inherent in natural tooth structure that resist root fracture include the strength of the inorganic (enamel, dentin) component, the elasticity of tooth structure resulting from its organic component, and the amount of remaining tooth structure present. Factors inherent in the cementation of endodontic dowels that promote root fracture include a decrease in moisture content and elasticity of the remaining tooth structure after removal of pulpal contents, discrepancies in the amount of remaining dentin after dowel space preparation as a result of morphologic differences in root structure, and the release of residual stresses resulting from lateral condensation of gutta-percha, hydraulic pressure during cementation, or discrepancies in size between the dowel and reamer system.²⁵ Fac. of Grad. Studies, Mahidol Univ.

Bex et al.²⁶ investigated the effect of dentin-bonded resin post-core preparations on resistance to vertical root fracture. In their experiment, dentin surfaces were treated with an eight-step process described by Bowen et al.⁹⁹ in 1983 that uses the 6.8 % aqueous solution of ferric oxalate, the adduct of N[p-tolyl] glycine and glycidyl methacrylate (NTG-GMA), and the 5 % acetone solution of the addition reaction product of pyromellitic dianhydride and 2-hydroxyethyl methacrylate (PMDM). They concluded that the dentin-bonded resin post-core restorations provided significantly less resistance to failure than the restorations with cemented custom cast post-cores and that the dentin-bonded resin post-cores fractured in every instance before the root fractured. They mentioned that the integrity of the root was preserved in every instance, although the resin post-cores failed under significantly less stress than the teeth restored with cast posts and cores. Greater force was required to cause failure of the resin post as the cross-sectional area of the post increased.

Donald et al.¹⁰⁰ studied the influence of dentinal adhesives and a prefabricated post on fracture resistance of silver amalgam cores. They concluded that endodontically treated mandibular molars restored with silver amalgam cores were more resistant to fracture when an adhesive agent was used.

Saupe et al.¹⁰¹ in 1996, compared the fracture resistance between morphologic dowels and cores (custom cast post-cores) and a resin-reinforced dowel systems in the restoration of structurally compromised roots. Their results indicated that the resistance to masticatory load of a resin-reinforced post-and-core system was greater than that of a morphologic post-and-core procedure. They also mentioned that, when a bonded resin reinforcement post system was used on structurally weakened roots, there was no statistically significant difference between post-and-core restorations that used a ferrule and those without a ferrule.

It is often assumed that the higher incidence of failure for restored endodontically treated teeth is primarily related to the fragility of the remaining tooth structure.¹⁵ However, this may not be the only reason for the higher failure rate. An in vivo investigation of the pressoreceptive function of endodontically treated teeth by Randow and Glantz¹⁰² in 1986 indicated reduced tactile sensation with pulpless teeth. This altered pressoreceptive capacity may be a significant factor contributing to functional overload of pulpless teeth.

In addition to the possibility of root fracture and the methods for minimizing its occurrence, Baraban²³ suggested that attention should be directed to the possibility of fracturing of the post itself. Equally important to providing a strong post to resist fracture is the adjustment of the occlusion of the final restoration to functional harmony. This adjustment helps ensure that the forces transmitted to the restored tooth will be within a tolerable range.

There are several retrospective studies investigating the failure rates of endodontically treated teeth. Vire¹⁰³ emphasized that a greater number of endodontically treated teeth are lost because of restorative failure, and 59.4% of the failed teeth were prosthetic failures which were due primarily to crown fracture. Weine et al.³³ conducted a retrospective study of 138 teeth that were restored with tapered smooth posts and cores with complete or 7/8 cast ferrules and an onlay or cast precious metal crown. The overall success rate was 93.5%. They concluded that when tapered smooth posts are used according to commonly accepted guidelines retentive problems do not occur.

Many factors relative to post-and-core restorations have been investigated and analyzed. The "type of abutment" has been shown to play a role in the failure of endodontically treated teeth.³⁵Abutments to fixed partial dentures recorded a significantly higher failure rate than abutments to removable partial dentures. Randow and Glantz¹⁰² in 1986 also demonstrated a correlation between the failure rate of endodontically treated teeth and the type of abutment. They concluded that prosthetic restorations with inherently high tendencies to generate functional bending should be avoided when the remaining distal teeth are root filled.

Another important requirement for post materials is stiffness. The stiffness of the post (a combination of cross-sectional geometry and modulus of elasticity) is an extremely important characteristic. Insufficient stiffness of the post permits micro-movement of the core and distortion of the restoration at the margins during function or cement breakdown and recurrent caries. However, the post must not be so stiff that it cannot deform compatibly with the root as it bends under functional loads. The material selected for post fabrication should also have high yield strength and possess favorable fatigue properties.^{60, 104}

Some researchers suggested that a post should be resistant to corrosion because several reports have linked root fracture to the corrosion of the post-and-core.^{105, 106} There has been unreliable evidence of formation of corrosive products from posts because of the interaction of posts with fluids from the dentinal tubules or the electrolytic action of dissimilar metals used for the post and core. These corrosive products could potentially cause a volumetric change within the tooth that may lead to longitudinal cracks, root fracture and eventual tooth loss.¹⁵⁰ Although a theoretically possible mechanism of fracture, it is more likely that the corrosion occurred subsequent to root fracture rather than acting as the cause of the root fracture itself. Further studies are needed to answer this question definitively.

2.7 Post retention and stress

The superior retentive abilities of a parallel-sided post over a tapered post have been well demonstrated.⁶ Johnson and Sakumura³⁸ reported that a parallel-sided post resists tensile forces 4.5 times greater than tapered posts. Ruemping et al.¹⁰⁷ measured the maximal tensile and torsional forces sustained by four different designs of posts and reported that: 1) under tensile force, the threaded screw-in posts were significantly more retentive than the unthreaded posts; and 2) under torque, both the threaded screw-in and serrated posts were significantly more retentive than the source sustained posts.

By using photoelastic stress analysis, tapered posts performed favorably during cementation and prior to loading. However, during loading to simulate functional stresses, tapered posts demonstrated unfavorable stress distribution compared with cylindrical posts. These posts also developed a wedging effect with excessive shoulder stress concentrations at the junction of the root surface and the internal portion of the post.^{12, 60, 108} Higher apical stresses were demonstrated for the Para-Post Plus post (parallel-sided, serrated prefabricated post) whereas the threaded posts concentrated stress where they engaged the model through threads or flanges.⁴² The results of other studies indicated that threaded posts produced high levels of stress at the apex if they were screwed the entire length of the prepared canal, and cylindrical posts dispersed stresses more uniformly along the root.^{60, 104, 108-112} Yaman and

Thorsteinsson⁴³ suggested that stiffer core materials can shift the load from the apex to the coronal region.

Assif et al.¹¹³, in 1989 found no difference between post designs for teeth with a post and core covered by a complete crown with a 2 mm. ferrule margin on sound tooth structure when the crowns were subjected to loading. They suggested that the complete crown may be the great equalizer, because it tended to change the distribution of forces to the root, post and core complex, with the post characteristics becoming insignificant.

Hunter et al.¹¹⁴ investigated the effects of post placement on endodontically treated teeth by using two-dimensional photoelastic stress analysis. Their results supported the theory that a post with a moderate diameter and length substantially reduced stress, if considerable enlargement of the root canal had occurred. They also reported that increasing post length up to two thirds the length of the root reduced stresses at the cervical region, and post placement beyond two thirds of the root depth did not further decrease cervical stresses but usually increased stresses in the apical region.

Walton et al.¹¹⁵ evaluated apical root strain as a function of post extension into a composite resin core. Their results indicated a statistically significant decrease in strain when 1 mm. of composite resin covered the head of the post. However, they suggested that the difference may not be clinically significant. By using strain gauges, Ross et al.¹¹⁶, compared the strains generated during placement of five endodontic posts. They reported that maximal strains accompanying placement of the Kurer Fin Lock Anchor and the Radix Anchor posts (threaded posts) were significantly higher than those induced by placement of the Flexi-Post post, V lock post and Para-Post Plus post. They also suggested that when the threaded posts were allowed to contact the apex of the prepared channel, high strain resulted.

The diameter of a post has an effect on retention, strength and ability to resist distortion. The smaller the diameter of a post, the more it will be displaced with or without accompanying distortion or fracture. Increasing the diameter of 5.0-mm. long parallel-sided posts by 0.25 mm. increased retention by 53%.¹¹⁷ Nevertheless, Standlee et al.⁶ suggested that variations in post diameter had no significant effect on retentive ability.

Because the strength of an endodontically treated tooth is directly related to the bulk of dentin remaining, the diameter of the post should be minimized and the bulk of the dentin maximized.¹² Mattison¹¹⁰ investigated cast-gold posts of different diameters to analyze the stress distribution and concentration in the dentin. The results of his study suggested that stresses generally increased as post diameter increased.

Post-and-core restorations rely on cementation for retention to the teeth. Cement is brittle and may disintegrate under continued cycling loading.¹¹⁸ Posts can be cemented with zinc phosphate cement, polycarboxylate cement, glass ionomer cement, or resin cements. Zinc phosphate cement is a traditional dental luting agent with a long and satisfactory clinical history. It provides retention through interlocking of micro-mechanical undercuts in the tooth structure and restorative materials. The retention is sufficient for well-designed posts, cores, and coronal restorations but does not equal the retention reported in vitro for chemically adhesive resin cements.^{119, 120}

According to a study by Standlee et al.⁶ in 1978, the greatest single factor influencing retention of the posts was the design used. The most retentive posts were threaded, parallel-sided posts screwed into tapped channels, and smooth-sided tapered posts were the least retentive. Another major factor in retention was the length of embedment in dentin. An increase in post embedment depth usually corresponded to increased retention, so post retention was proportional to post length.¹⁰⁷ Increasing the post length from 5.0 to 8.0 mm. increased the retention by about one and one-half times.^{6, 117} Commonly accepted guidelines for post length in a tooth with normal periodontal support include two thirds of the length of the canal, the coronal length of the tooth, and half of the bone-supported length of the root.³⁷ Greater leverage is exerted when the post is shorter than the clinical crown length, and this unfavorable leverage can also predispose the root to fracture.¹⁰⁴ The final length of the post in a periodontally healthy tooth is limited by two major variables, the root morphology and the need for sufficient apical seal.¹⁵ Mattison et al.¹²¹ suggested at least 5 mm. of gutta-percha is necessary for an adequate apical seal, and the mechanical method was the most desirable approach for gutta-percha removal during post preparation. Other studies have demonstrated that no effect on the apical seal occurred if 4 mm. or more of gutta-percha remained in the apical portion of the canal.¹²²⁻¹²⁴ The root should have at least 1 mm. of tooth structure remaining around the post in all directions in order to
resist fracture or perforation.⁴⁴ Endodontically treated teeth with root curvatures and root concavities require alteration of the length of posts to eliminate thin radicular walls or root perforations.¹⁵

The effect of cement type was significant only with tapered posts. For the other post designs, cement type had no significant effect on retentive capacity.⁶ Other previous studies on retention of Para-Post dowels cemented with zinc phosphate cement, zinc polycarboxylate cement, glass ionomer cement and composite resin cement reported no correlation between the retention and cement type.¹²⁵⁻¹²⁷ Millstein et al.¹²⁸ concluded that there was no substantial difference between the retention added to screw posts by either resin cement or zinc phosphate cement. There are other studies that have indicated higher retention for prefabricated posts cemented with resin-based cements in comparison to posts cemented with conventional cements.^{119, 120} However; Cohen et al.¹²⁹ reported that the Filpost system achieved higher retention with zinc phosphate cement than it did with resin cement.

Assif and Bleicher¹³⁰ in 1986 reported that when composite resin cement was used, the adaptation of the post to the canal was not crucial. Changes in the thickness of the composite resin film up to 500 microns did not decrease retention. Variations in the diameter of the posts also did not affect the retention. They also suggested that the removal of tooth structure is unnecessary because the cylindrical preparation is formed in the composite resin. Standlee and Caputo¹³¹ supported the concept that resin-cemented posts can be retained similar to actively seated posts. They also investigated the effect of surface design on retention of posts cemented with a resin cement.¹³² Posts with transverse serrations or crosshatching were retained better than posts with longitudinal spirals or threads.

Attempts have been made to improve the retention of posts by using resin cements with a chemical pretreatment of the canal.^{119, 133-136} Removal of the smear layer by the use of 17% EDTA followed by a solution of 5.25% NaOCl revealed significant increases in retention when compared with conventional methods, even with shorter posts.¹³⁵ This result has been explained by the extension of cement into the open dentinal tubules. However, there are other studies that did not report improved retention with the use of EDTA.^{137, 138} The increasing use of composite resins as luting agents for prefabricated posts may enhance retentive applications for

dentinal bonding agents. A significant increase in retention could result in more conservative post preparations and fewer root fractures.

Chapman et al.¹²⁶ evaluated the effect of bonding agents on retention of posts. They reported that added retention may occur with the use of a bonding agent; nevertheless retention depended on the particular composite resin used and on careful attention to the technique applied. El-Mowafy and Milenkovic¹³⁹ reported that Para-Post posts cemented with Prisma Universal Bond 3/Biomer or with Scotchbond Multi-Purpose/Resiment systems required significantly greater separation forces than posts cemented with any of the other cementation systems. Mendoza et al.¹⁴⁰ demonstrated that roots with extremely flared canals in which the posts were cemented with Panavia resin cement were significantly more resistant to fracture than those where zinc phosphate cement was used. Mendoza and Eakle¹⁴¹ investigated the retention of posts cemented with various dentinal bonding cements. Their results indicated that C&B Metabond resin cement was the most retentive. No difference in retention was recorded between Ketac-Cem and Panavia cements. All-Bond 2 cement was the least retentive of the cements studied. Standlee and Caputo¹³¹ also reported that resin cement with the C&B Metabond regimen was the most retentive when compared to Boston Post regimen and Unity Post regimen.

When cementing posts with composite resin cement, conditioning of root canals prior to post cementation has been suggested.^{142, 143} The procedures should follow manufacturer's instructions for the cement systems and dentinal bonding agents used. In one study, Scotchbond dentinal adhesive gave significantly better results, regardless of the cleansing solution used.¹⁴² Tagami et al.¹⁴⁴ recommended Superbond C&B as both an enamel- and dentin-bonding agent. However, bond strengths varied depending on the dentinal bonding agent used.^{141, 144}

Tjan and Nemetz¹⁴⁵ in 1992 investigated the effect of residual eugenol in the root canal on the retention of Para-Post posts cemented with Panavia EX composite resin cement. Eugenol, which is also a component of most commercially available root canal sealers, significantly reduced the retention of posts luted with Panavia composite resin cement. Irrigation of the post space with 95 % ethanol restored the retention. Maryniuk et al.¹⁴⁶ studied the effects of canal lubrication prior to making a custom acrylic resin post pattern on retention of cemented posts with zinc phosphate cement. Their study indicated that residual lubricant greatly reduced the retention of cemented cast posts. Water rinsing alone did not remove the lubricant, but a cavity cleaner solvent did eliminate the lubricant effectively.

Successful endodontic treatment for pulpal pathosis will result in an asymptomatic tooth with a sealed canal. Restorative treatment for the pulpless tooth should provide protection against fracture and the needed resistance against masticatory stress to allow the tooth to resume normal function. There is still need for a post-core system to perform harmoniously with the remaining tooth structure under function. No single restorative method or material will be successful in every situation. Treatment should be flexible and based on scholarly diagnostic considerations supported by current research and technical information.

2.8 Alloy used in dentistry

Cast alloys are those which are heated to form a liquid and then the molten metal is casted into a mould where it solidifies. Complicated shapes can be produced in this way. Current System for Classifying Dental Alloys was defined according to International standard. ISO22674:2006; Dentistry - Metallic materials for fixed and removable restorations and appliances. As shown in table below:

Туре	Descriptions
0	Intended for low stress bearing single-tooth fixed restorations, e.g. small veneered one-
	surface inlays, veneered crowns.
1	Intended for low stress bearing single-tooth fixed restorations, e.g. veneered or
	unveneered one-surface inlays, veneered crowns.
2	Intended for single tooth fixed restorations, e.g. crowns or inlays without restriction on
	the number of surface.
3	intended for multiple unit restorations, e.g. bridges.
4	Intended for appliances with thin sections that are subject to very high forces, e.g.
	removable partial dentures, claps, thin veneered crowns, wide-span bridges or bridges
	with small cross-sections, bars, attachments, implant retained superstructures.

Table 2-1. Metallic material classification

Table 2-1. (cont.) Me	tallic material	classification
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Туре	Descriptions
5	Intended for appliances in which parts require the combination of high stiffness and
	strength, e.g. thin removable partial dentures.

2.9 Dental copper-aluminium alloys

In behavior noble metal alloys have a corrosion resistant but cost is higher, while the alloys containing copper have a lower corrosion resistance and cost is reasonable.¹⁵¹Also to possessing a low cost, this alloy can be cast using the same basic procedure as that employed with gold-based alloys. Alloyed with elements such as iron, nickel, manganese and tin, consisting principally of copper and aluminum at a proper ratio.¹⁵² They show in fact quite a good color match to gold, and keep a surprising brilliance in the oral environment.

The copper-aluminium alloys are a family of copper-base alloys containing aluminium as the most important alloying element approximately 5 wt % to 11 wt %; some are having additions of iron, nickel, manganese, tin or silicon which range from 5 wt % to 14 wt %.¹⁵³ They gives respond to alloys which are harder and stronger than copper and show sufficient ductility to resist fatigue and well-known for their elevated outstanding corrosion resistance under a wide range of service conditions. The corrosion resistance depends upon the formation of protective film or layer of corrosion products which prevents or substantially slows down the rate of attack. The aluminium and nickel content of copper-aluminium alloys imparts the ability to form, very rapidly, a protective film which is highly protective and is not prone to localized breakdown and consequent pitting in the presence of chlorides.¹⁵³ The majority of additions of alloying elements develop from these properties.

2.10 An ideal post system

An ideal post system should have the following features:

- Maximum retention with little removal of dentin
- Distribution of functional stresses evenly along the root surface

- Esthetic compatibility with the definitive restoration and surrounding tissue
- Minimal stress during placement and cementation
- Resistance to displacement
- Good core retention
- Easy retrieve-ability
- Material compatibility with core
- Ease of use
- Reasonable cost

2.11 Toxic reactions to nickel-containing dental alloys

The suggestions of nickel-containing alloys in oral conditions do not fulfill the conditions for nickel ions to exhibit any carcinogenic effects.¹⁷⁰

The nickel-containing is found in many alloys used in the practice of dentistry. These alloys have a long-lasting history of successful use in dentistry, with no significant reports of biological effects.¹⁷¹ There is no evidence of carcinogenicity associated with the intraoral use of nickel-containing dental alloys.¹⁷²

Nickel compounds are universal and are consumed as part of a normal diet from foods such as vegetables, with the daily intake estimated to be 100 to 600 μ g/day.¹⁷³ Gjerdet NR et al., reported that the effect of elements elution on human epithelial cells of non-precious dental casting alloys containing up to 84 wt % Ni did not reach cytotoxic levels.¹⁷⁴

2.12 Suggestion

There is a need for long term clinical evaluation of both metallic and nonmetallic post systems to allow a definitive recommendation of either of them.

Until such time, metallic posts continue to be the standard for most situations because they have verified the valuation for the time.

CHAPTER III MATERIALS AND METHODS

3.1. Materials

- 3.1.1 Copper (Wako Pure Chemical Industry, Ltd., Osaka, Japan)
- 3.1.2 Aluminium (Wako Pure Chemical Industry, Ltd., Osaka, Japan)
- 3.1.3 Nickel (Wako Pure Chemical Industry, Ltd., Osaka, Japan)
- 3.1.4 Graphite crucible for Linn[®] HFS 3 (Linn High Therm GmbH, Eschenfelden, Germany)
- 3.1.5 Acetal copolymer rod: Ertacetal[®] C (Gilbert Curry Industrial Plastics Co Ltd, Coventry, UK)
- 3.1.6 Silicone: Dow corning[®] Mold life extender (Dow corning Corp, MI, USA)
- 3.1.7 PMMA Sheet: SumipexTM TL (Sumitomo Chemical Co.,Ltd., Chiba, Japan)
- 3.1.8 Green quick sprue wax (Kerr Corporation, CA, USA)
- 3.1.9 Blue inlay wax (Kerr Corporation, CA, USA)
- 3.1.10 Silver Epoxy Resin: SPI Fast Setting Conductive Silver Epoxy (SPI Supplies and Structure Probe, Inc., PA, USA)
- 3.1.11 Transparent epoxy adhesives: PATTEX[®] (Henkel AG & Co., Düsseldorf, Germany)
- 3.1.12 Cristobalite investment (Shofu Inc, Kyoto, Japan)
- 3.1.13 Separating disc: Major K (Luoyang Beiyuan Special Ceramics Co.,Ltd., Henan, China)
- 3.1.14 Stone and diamond bur: GRS (Glendo Corp, KS, USA)
- 3.1.15 Epoxy resin: Epo-Kwick[®] Fast Cure Epoxy Kit (Buehler GmbH, Düsseldorf, Germany)
- 3.1.16 Abrasive Discs: 10" FEPA P400, P800, P1200 BuehlerMet[®] II (Buehler GmbH, Düsseldorf, Germany)

- 3.1.17 Abrasive Discs: 10" FEPA P1500, P2500, P4000 MicroCut[®] (Buehler GmbH, Düsseldorf, Germany)
- 3.1.18 Lactic acid (Merk& Co., Inc., Darmstadt, Germany)
- 3.1.19 Sodium chloride (Merk& Co., Inc., Darmstadt, Germany)
- 3.1.20 Sodium sulfide (Merk& Co., Inc., Darmstadt, Germany)
- 3.1.21 L929 cells: NCTC clone 929, ATCC[®] Lot no. 2869501 (American Type Culture Collection, VA, USA)
- 3.1.22 Neutral red: 101369 Neutral red, C.I. 50040 (Merck KGaA, Darmstadt, Germany)
- 3.1.23 Trypan blue stain: 15250-061 Trypan blue stain, 0.4% (Gibco, Invitrogen Corporation, CA, USA)
- 3.1.24 Dulbecco's Modified Eagle Medium: DMEM Gibco[®] (Gibco, Invitrogen Corporation, CA, USA)
- 3.1.25 Agar: Calbiochem[®] 12177Agar (Merck KGaA, Darmstadt, Germany)
- 3.1.26 Cell cultured dish: 100 mm x 20 mm Corning[®] Cell Culture Treated Dish (Corning Incorporated, NY, USA)
- 3.1.27 Cell culture flask: 75 cm² Corning[®] Growth area flask (Corning Incorporated, NY, USA)
- 3.1.28 Fetal bonvine serum: HyClone Bovine Growth Serum, U.S. Origin (Thermo Scientific HyClone, UT, USA)
- 3.1.29 Trypsin tissue cultured grade: 0458 Trypsin (Amresco Inc., OH, USA)
- 3.1.30 Positive control: Polyurethane film-ZDEC (RM-A) (Hatano Research Institute, Food and Drug Safety Center, Kanakawa, Japan)
- 3.1.31 Negative control: Thermanox[®] (TMX) sterile, polyolefin polymer coverslip (Nalge Nunc International, Thermo Fisher Scientific, NY, USA)
- 3.1.32 Power Glue[®] (Cyanoacrylate adhesive, Alteco Chemical PTE LTD.)

3.2 Apparatus

- 3.2.1 Analytical Balance: Precisa[®] 262 SMA-FR (Precisa Instruments AG, Dietikon, Switzerland)
- 3.2.2 High frequency casting / melting unit: Linn[®] HFS 3 Vacutherm (Linn High Therm GmbH, Eschenfelden, Germany)
- 3.2.3 Differential scanning calorimeter: Rigaku[®] DSC8270 Thermal Analyzer (Rigaku Corporation, Tokyo, Japan)
- 3.2.4 d*TREK 9.9.3 and dtrek2jpeg software (Rigaku Corporation, Tokyo, Japan)
- 3.2.5 Vacuum wax melting injector: Injector 012 (Oma Tools and Machinery, Bangkok, Thailand)
- 3.2.6 Vacuum Mixing device: Vacuret-S[™] (REITEL[®] Feinwerktechnik GmbH, Bad Essen, Germany)
- 3.2.7 Preheating furnace: BEGO[®] Miditherm 200MP (Bremer Goldschlägerei Wilh. Herbst GmbH & Co. KG, Bremen, Germany)
- 3.2.8 Microflame melting torch for gas/oxygen: BEGO[®] Multiplex (Bremer Goldschlägerei Wilh. Herbst GmbH & Co. KG, Bremen, Germany)
- 3.2.9 Benchtop centrifugal casting machine for flame melting: BEGO[®]
 Fundor T (Bremer Goldschlägerei Wilh. Herbst GmbH & Co. KG, Bremen, Germany)
- 3.2.10 Micro-drive Handpiece: Kavo[®] EWL[™] 4990 (KaVo Dental GmbH, Warthausen, Germany)
- 3.2.11 Microhardness tester: FM-700 (Future-tech Corp., Kanakawa, Japan)
- 3.2.12 Automatic indentation measuring system: AR90 (Future-tech Corp., Kanakawa, Japan)
- 3.2.13 Universal testing machine: Instron[®] Model 5566 (Instron Corporation, Buckinghamshire, UK)
- 3.2.14 Materials Testing Software: Merlin[™] (Instron Corporation, MA, USA)

- 3.2.15 Dial Caliper Gauge: 0.01mm Resolution Mitutoyo[®] 209-609 (Mitutoyo Corp., Kanagawa, Japan)
- 3.2.16 Atomic absorption spectrometer: Varian SpectrAA-200 (Agilent Technologies Inc., CA, USA)
- 3.2.17 AA Instrument Software (Agilent Technologies Inc., CA, USA)
- 3.2.18 Ultrasonic cleanser: TRU-SWEEPTM Crest 275D (Crest Ultrasonic Corp, NJ, USA)
- 3.2.19 Potentiostat/Galvanostat: Solartron[™] 1260A (Solartron
 Analytical, AMETEK Advanced Measurement Technology, Hampshire, UK)
- 3.2.20 Scanning electron microscope (JSM-5410 LV), [JEOL Ltd., Tokyo, Japan)
- 3.2.21 Three electrodes double-walled corrosion cell (Fisher Scientific Inc., PA, USA)
- 3.2.22 Reference Electrodes: AccumetTM Ag/AgCl, 13-620-53 (Fisher Scientific Inc., PA, USA)
- 3.2.23 Counter Electrode: PT005150, 99.95% Platinum wire, dia. 1 mm (Goodfellow Cambridge Ltd., Huntingdon, UK)
- 3.2.24 Electrochemical measurements software: CorrWare[®] and CorrView[™], Version 3.20b (Scribner Associates, Inc., NC, USA)
- 3.2.25 Incubator: Memmert[®] 600 (Memmert GmbH & Co.KG, Schwabach, Germany)
- 3.2.26 pH meter: OrionTM 710 (Thermo electron corp., MA, USA)
- 3.2.27 Dipping device: TC 400 (Medical & environmental equipment research lab., KMIT Lad-krabung, Bangkok, Thailand)
- 3.2.28 Spectrophotometer: HunterLab[®] Color Flex[™] 4510 (Hunter Associates Laboratory, Inc., VA, USA)
- 3.2.29 HunterLab[®] Universal Software (Hunter Associates Laboratory, Inc., VA, USA)
- 3.2.30 CO₂ Laboratory Incubator: Thermo Forma (Forma Scientific Inc., OH, USA)

- 3.2.31 Inverted microscope: Nikon Eclipse TS100/TS100F (Nikon Corporation, Tokyo, Japan)
- 3.2.32 Micro flow Advanced bio safety cabinet: (Bioquell UK Ltd., Hampshire, UK)
- 3.2.33 Temperature controlled water bath: Memmert (Memmert GmbH & Co.KG, Schwabach, Germany)

3.3 Methods

This study is separated into 4 parts.

Part 1

- 1. To prepare the aluminum-copper-nickel alloy with varying proportions of Al and Ni for dental post-and-core application.
- 2. To evaluate thermal properties of the experimental alloy.

Part 2

- 1. To evaluate surface hardness of the experimental alloy.
- To evaluate Tensile strength, Elastic of modulus, Elongation and 0.2% Proof strength of the experimental alloy.

Part 3

- 1. To evaluate corrosion resistance and electrochemical behavior (Potentiodynamic polarization) of the experimental alloy.
- 2. To evaluate tarnish resistance (Static and Dynamic immersion) of the experimental alloy.

Part 4

To evaluate biocompatibility (Agar diffusion) of the experimental alloy.

Part 1: Alloy preparation and study of melting range of the experimental alloy at different composition.

3.3.1 Experimental alloy processing.

Sixteen different groups of copper alloys with varying proportions of Al and Ni were alloyed corresponding to Table 3-1. The experiment alloy numbers were run as shown in Figure 3-1. This numbers and order in the diagram would apply in all the studies.

Alloys	Contents				
No.	Ni (weight %)	Al (weight %)	Cu (weight %)		
1	-	3	97		
2	-	6	94		
3	-	9	91		
4	-	12	88		
5	2	3	95		
6	2	6	92		
7	2	9	89		
8	2	12	86		
9	4	3	93		
10	4	6	90		
11	4	9	87		
12	4	12	84		
13	6	3	91		
14	6	6	88		
15	6	9	85		
16	6	12	82		

Table 3-1. The element compositions of the experimental alloys.

The raw materials were used high purify (>99.95%) elements (Wako Pure Chemical Industry Ltd, Osaka, Japan). Each group was weight approximately 35 g using analytical balance (Precisa 262 SMA-FR) and meltdown within a 25 mm diameter cylindrical graphite crucible sleeve using induction casting unit (Linn HFS 3) which casting arm is combined with vacuum tube. The vacuum operation was approximately 50 mbar during process. The melted alloys were bench cool within the crucible covered with graphite cap.

3.3.2 Measurement of Melting Range

The melting range of alloys was determined using Thermal analyzer Differential Scanning Calorimeter (Rikaku DSC8270).

Approximately 1 gram of each sample was heated at a rate of 10 $^{\circ}$ C/min extended to 1300 $^{\circ}$ C. The measurements were made using "d*TREK" software on two samples for each group.

Part 2: Study of the physical properties and mechanical properties of the experimental alloy.

3.3.3 Preparation of test specimens for physical properties and mechanical properties.

The hardness, tarnish and corrosion preformed patterns were pre-cutting PMMA sheet (Sumipex[®] TL) in dimensions of 10 mm X 10 mm X 2 mm and 34 mm X 13 mm X 1.5 mm. The tensile dumbbell preformed patterns with a screw thread at the end, size 3 mm in diameter and 42 mm long with 18 mm gauge length in accordance with ISO standard (ISO 22674:2006) were constructed using acetal copolymer rod (Ertacetal[®] C) combined with the injection of molten blue inlay wax (Kerr[®]) into a split-silicone mold (Dow corning[®]).

The tensile, tarnish and corrosion specimen patterns for their castings are shown in Figure 3-2.



Figure 3-2. Specimen pattrens assembly.

Those patterns were invested in cristobalite investments (Shofu[®]) using vacuum mixing device (Vacuret-STM) on ring procedure. Heated up the ring using preheating furnace (BEGO[®] Miditherm 200 MP) and then casted by gas-oxygen torch using bench top centrifugal casting machine (BEGO[®] Fundor T). After cooled down at room temperature, all specimens were carefully separated from sprue. The excess nodules or fins were removed using a separating disc (Major K) and stone bur (GRS). Specimen with visible shrinkage defects or porosity was discarded from this study.

3.3.4 Determination of surface hardness

Four of six with dimension of 10 mm-square and 2 mm-thick test specimens for physical/mechanical properties from each group were mounted in epoxy resin (Epo-quick®). After that the surface of mounted specimen was prepared with metallographic grinder/polisher (Rotopol-21) using standard metallographic procedure, starting from FEPA P 200 to FEPA P 1000 silicon carbide paper (Buehler) and finished with 1 µm diamond paste. The test surface should be smooth and even, free from oxide scale, foreign matter, and completely free from lubricants. Each specimen was placed on micro-hardness tester (Future-tech® FM-700), and then the Vickers diamond indenter was brought into contact with the test surface. The load was applied in a direction perpendicular to the specimen surface at 1Kgf, for 15 seconds without shock or vibration. Five reading were taken from a center and corner regions of each specimen. And the distance between the center of several indentations and the edge of the test specimen were not less than 2.5 mm. All data was collected and analyzed statistically.

3.3.5 Determination of tensile strength, proof strength of non-proportional extension and percentage elongation properties after fracture.

Six specimens of each group will be loaded in tension on universal testing machine (Instron model 5566) at the crosshead speed of 1.5 ± 0.5 mm/min until fracture occurred.

The ultimate tensile (Rm), 0.2% proof strength of non-proportional extension (Rp_{0.2}), elastic modulus (E) and the percentage elongation (A) after fracture of each group will be determined in accordance with ISO 6892-1:2009 and ISO 22674:2006.

Part 3: Study of the corrosion resistance, electrochemical behavior and tarnish resistance of the experimental alloy.

3.3.6 Study of the corrosion resistance and electrochemical behavior.

The experimental alloys will be subjected to tests for the determination of tarnish and corrosion following general guidance for corrosion tests presented in ISO 10271:2001, corrosion tests methods for dental metallic materials and ISO 22674:2006, metallic materials for fixed and removable restorations and appliances. The information of the kind and amount of metal ions leached from the alloy will be recorded and analyzed. The following tests will be applied to each group of alloy.

3.3.6.1 Electrochemical test (Potentiodynamic polarization

test)

Four specimens of ten millimeter square and two millimeter thick of each alloy groups were combined with stainless steel nuts using silver conductive epoxy glue (SPI). And then the whole construction will be covered with an electrically insulating epoxy binder (UHU Quickset) and embedding in Buehler epoxy resin (Epo-quick). In this manner the stainless steel bolt working electrode was constructed. The exposed surface of embedded specimens were prepared with metallographic grinder/polisher (Struers Rotopol-21) using standard metallographic procedure, starting from FEPA P 200 to FEPA P 1000 silicon carbide paper (Buehler) and finished with 1 μ m diamond paste (Buehler). Consider the exposed area of the test specimen was approximately 1 cm². The specimens were finished in distilled water using ultrasonic cleanser (CP100HT).

The polished surfaces were observed with a light microscope (Nikon Optiphol 2) at X 50 for cracks and spaces on the specimen/resin interface. Replace the sample if any of them were found. The specimens were store in distilled water until transfer to corrosion cell.

The 0.9 % sodium chloride electrolyte with the pH value of 7.4 ± 1 was prepared using the procedure according to ISO 10271:2001. The experiments were

carried out in three electrodes double-walled corrosion cell using silver/silver chloride (Solartron Accumet) served as a reference electrode (RE), one millimeter in diameter platinum wire (PT005150) served as a counter electrode (CE) and the alloy specimens served as a working electrode (WE). The electrochemical cell was 37 °C conditioned using circulated temperature controlled water bath (Memmert) and connected to a Potentiostat/Galvanostat (Solartron 1260A). The open circuit potential and potentiodynamic technique was selected for this study. The Corrosion cell setup diagram is shown in figure 3-3. The measurements were controlled and computed via electrochemical software (CorrWare and CorrView Version 3.20b).

3.3.6.2 Open-circuit potential measurement

An *in-vitro* Potentiodynamic polarization test was employed to evaluate the electrochemical behavior of the experimental alloys. Bubbled nitrogen gas through the electrolyte at a rate of 100 cm³/min for at least 30 minutes and then streamed into the cell. The open circuit potential will be registered for 2 hours. Record the open circuit potential (E_{ocp}) in mV (SCE) versus time curve.



Figure 3-3. Corrosion cell set up

3.3.6.3 Potential measurements (Anodic polarization)

The potentiodynamic will be scanned 5 minutes after finishing the OCP measurement at a rate of 1 mV/sec from the minus 150 mV of the E_{ocp} up to a potential of +1000 mV. Record the curve of potential versus logarithm of current density.

3.3.7 Study of the tarnish resistance of the experimental alloy.

The specimens will be subjected to tests for the determination of tarnish and corrosion following general guidance for corrosion tests presented in ISO 10271:2001 and ISO 22674:2006.

3.3.7.1 Static immersion test

Two of six specimens with dimension of 34 mm X 13 mm X 1.5 mm for static immersion test from each group, will be separated cleanly from the sprues, freed of casting beads and then be grit blasted. At least 0.1 mm from all sides of specimen will be removed using standard metallographic procedures, ending with FEPA P 1200 wet silicon carbide paper. After that, any residual abrasive, oil or grease will be removed. Any test specimens with visible defects will be replaced.

The surface area of each test specimens will be measured and recorded to the nearest 0.1 cm^2 , and then specimens will be ultrasonically in ethanol for 2 min, rinse and dry with water and oil-free compressed air. For each specimen will having a total surface of approximately 10 cm². The aqueous solution comprising 0.1 mol/l lactic acid and 0.1 mol/l sodium chloride with the pH value in range of 2.2 to 2.4 will be prepared immediately before use.

The glass laboratory beaker approximately 16 mm diameter X 16 mm will be apply to contain the aqueous solution as a result to avoid absorption of trace elements on the surface of the container. The volume of solution in each beaker will be completely covered the specimen, approximately 1.3 ml /cm² of the test specimen surface area. Each test specimens will be suspending by hanging on nylon strings in a separate container at 37 ± 1 °C for 7 days. The beaker will be sealed tightly to prevent evaporation. After 7 days, all specimens will be removed and the test solutions will be collected and analyzed separately using atomic absorption spectrometry (Varian SpectrAA-200). The values of each observed elements present will be recorded in μ g /cm² per seven days.

3.3.7.2 Tarnish test (Cyclic immersion)

Two of six with dimension of 10 mm-square and 2 mm-thick test specimens for physical/mechanical properties of each alloy groups, will be mounted in epoxy resin (Epo-quick). After that the surface of mounted specimen will be prepared with metallographic grinder/polisher (Struers Rotopol-21) using standard metallographic procedure, starting from FEPA P 200 to FEPA P 1000 silicon carbide paper and finished with1 μ m diamond paste. Use fresh paper for each alloy. Clean surfaces ultrasonically for 2 minutes in distilled water. Rinse with distilled water and dry with oil and water-free compressed air. The test surface should be smooth and even, free from oxide scale, foreign matter, and completely free from lubricants. One of the two mounted specimen will be position on dipping device (KMIT TC 400) which dip the test specimen into the tarnishing solution for 10 to 15 second every minute at 23 ±2 °C for 72 ±1 hours. The test solution shall be replaced every 24 ±1 hours. The tarnish solution comprising 0.1 mol/l sodium sulfide will be prepared immediately before use.

After 72 ± 1 hours remove the specimen from dipping device, rinse it thoroughly with distilled water, dip it in ethanol, and then dry with oil-free compressed air. The specimen was evaluated the changed effect from the color difference (ΔE^*), values of each treated and untreated specimens using spectrophotometer (HunterLab Color Flex 4510).

Part 4: Study of the bio-compatibility of the experimental alloy.

3.3.8 Agar diffusion test (Agar overlay test)

Standards for the biocompatibility of dental alloys are still to be determined. The ISO recommends cytotoxicity as a screening test for dental materials. The goal of all biocompatibility testing is to simulate conditions that would exist in the oral environment. The term biocompatibility refers to the ability of a material to be in contact with living tissue and not cause toxic or injurious effects.

The experimental alloys were subjected to *in vitro* tests of cytotoxicity following general guidance for *in vitro* cytotoxicity tests presented in ISO 10993-5:1999, and other tests required for implant devices indicated in ISO 7405:2008.



Figure 3-4. Cell cultured dish and specimen location

Agar overlay technique was used as a biocompatibility test. The American Type Culture Collection L929-3T3 was used for cell line. The monolayer of cell line suspended in 3% agar to obtain a final concentration of approximately 3 x 10^5 cells/ml was prepared in the 90 mm-diameter culture plate. Six specimens from each alloy group were cut from the sprue with 5 mm in diameter and 1 mm thick. All sides of specimen were removed using FEPA P 1200 wet silicon carbide paper. The specimens were ultrasonically in ethanol for 2 min, rinsed and dried with distilled water and oil-free compressed air. Applied 10 milliliter of L929 cell with intensity of 2.5 x 10^5 cells per cubic centimeter in completed cultured DMEM medium into 90 mm petri dish. Incubated the dish in a CO₂ incubator at 37°C, 95% relative humidity and 5.0% carbon

dioxide for 24-hour. The cell style was a single layer (monolayer) and then taking out the cultured medium. Filled in the cultured dish with new more 45°C of agar cultured medium with contains completed 2x DMEM medium and 3% agar. And so left the cultured dish until agar cultured medium become stiff and then dyed with 1% neutral red.

Divided the area within the dish into 4 parts of testing area, and so applied positive (polyvinyl carbonate) and negative (Whatman AA disc) control specimen placed on the surface of the agar cultured medium on the right and left upper part, respectively (Figure 3-4), the bottom two sections provided 5 mm diameter \times 1 mm of experimental alloy dishes. And then incubated in a CO₂ incubator at 37°C, 95% and 5.0% carbon dioxide for 24-hour. After that the cultured dish was taken to evaluate a "*decolorization index*" and a "*lysis index*" using the description in Table 3-2 and 3-3.

The decolorization zone from the rim of the specimens, and cell lysis will be recorded. The ratio of the decolorization zone and the cell lysis ratio were recorded and so calculated as a "cell response" using the description in Table 3-4 to interpret the cytotoxicity (Table 3-2 and 3-3).

Table	3-2.	Narration	of	deco	loriza	tion	index

Decolorization	Description
index	
0	No detectable decolorization zone around or under specimen
1	Decolorization zone limited to area under specimen
2	Decolorization zone extends less than 0.5 cm beyond specimen.
3	Decolorization zone extends 0.5 cm to 1.0 cm beyond specimen.
4	Decolorization zone extends greater than 1.0 cm beyond specimen but does not involve entire dish.
5	Decolorization zone involves entire dish.

Apiwat Rittapai

Lysis index	Description
0	No observable cytotoxicity.
1	Less than 20% of the decolorized zone affected.
2	20% to < 40% of the decolorized zone affected.
3	40% to < 60% of the decolorized zone affected.
4	60% to < 80% of the decolorized zone affected.
5	Greater than 80% of the decolorized zone affected.

Table 3-3. Narration of lysis index

Table 3-4. Narration of cell response and interpretation

Lysis index	Description
0	No observable cytotoxicity.
1	Less than 20% of the decolorized zone affected.
2	20% to < 40% of the decolorized zone affected.
3	40% to < 60% of the decolorized zone affected.
4	60% to < 80% of the decolorized zone affected.
5	Greater than 80% of the decolorized zone affected.

CHAPTER IV RESULTS

4.1 Alloy preparation and study of the solidus, Liquidus point and melting range of the experimental Cu-Al-Ni alloy.

According to the experimental design in this study, the copper was alloyed with Nickel and Aluminum in various ratios. The experiment alloy numbers were run as shown in Figure 4-1. This numbers and order in the diagram would apply in all the results shown below; otherwise, recommended differently.

		E	Experiment alloy group number						
\uparrow	12	4	8	12	16				
	9	3	7	11	15				
	6	2	6	10	14				
Al (Weight %) 3	1	5	9	13				
Cu Balance		0 Ni (Weight %)	2	4	6 →				

Figure 4-1 The diagram of the experiment alloy design

The mean and standard deviation of the Solidus and Liquidus point as well as the melting range of the experimental alloys demonstrated in Figure 4-2 to 4-4.

Figure 4-5 showed the chart of solidus and liquidus point of the experimental alloy in order from alloy number 1 to 16 and trend line when the content of auminum was constant.

			Solidus p	ooint (°C)		
\uparrow	12	4 1031.5 (0.5)	8 1032.1 (0.1)	12 1040.9 (0.3)	16 1048.6 (0.2)	
		3	7	11	15	maximum
	9	1022.5 (0.9)	1024.8 (0.1)	1030.8 (0.0)	1020.8 (0.8)	^
	6	2 1014.7 (0.4)	6 1032.8 (1.1)	10 1045.2 (0.6)	14 1044.7 (0.1)	^ minimum
Al (Weight %)	3	1 1032.3 (1.2)	5 1057.4 (0.6)	9 1066.4 (0.4)	13 1069.2 (0.3)	
Cu Balan	ce	0 Ni (Weight %) –	2	4	ightarrow 6 (SD in p	arentheses)

Figure 4-2 Mean and SD of Solidus point of the experimental alloys

			Liquidus p	point (°C)		_
↑	12	4 1059.1 (0.4)	8 1061.0 (0.4)	12 1067.8 (1.2)	16 1077.6 (0.1)	
	9	3 1050.1 (0.1)	7 1052.3 (2.6)	11 1057.7 (0.1)	15 1058.3 (0.2)	maximum
	6	2 1056.5 (1.2)	6 1068.8 (1.1)	10 1077.2 (0.5)	14 1086.2 (1.5)	^ minimum
Al (Weight %)	3	1 1078.6 (0.1)	5 1092.5 (1.0)	9 1099.0 (1.7)	13 1113.4 (1.5)	
Cu Balan	ce	0 Ni (Weight %) -	2	4	\rightarrow (SD in p	arentheses)

Figure 4-3 Mean and SD of Liquidus point of the experimental alloys

			Melting r	ange (°C)		
↑	12	4 27.7 (0.1)	8 28.9 (0.2)	12 26.9 (0.9)	16 29.1 (0.1)	
	9	3 27.7 (0.8)	7 27.4 (2.5)	11 26.9 (0.1)	15 37.5 (1.0)	maximum
	6	2 41.7 (1.6)	6 36.0 (0.1)	10 32.0 (1.1)	14 41.5 (1.6)	 minimum
Al (Weight %)	3	1 46.3 (1.3)	5 35.1 (0.4)	9 32.7 (2.1)	13 44.2 (1.8)	
Cu Balan	ce	0 Ni (Weight %) -	2	4	$6 \rightarrow$ (SD in p	arentheses)

Figure 4-4 Mean and SD of melting range of the experimental alloys

As shown in figure 4-2 to 4-4, found that the solidus and liquidus points were increased as nickel content increased especially at the lower content of

aluminum; alloy number 1, 5, 9 and 13. The influence of aluminum content on the solidus and liquidus point was reversed in parabola shape that higher when the aluminum content was lowest or highest (3 wt % Al and 12 wt % Al) and decreased when the content was intermediate (6 wt % Al and 9 wt % Al) as showed clearly in Figure 4-4. The alloy number 13 (3 wt % Al, 6 wt % Ni) showed the highest solidus and liquidus point in this study. The lowest solidus and liquidus point in this study. The lowest solidus and liquidus point in this study was shown in alloy with the nickel content was 0 wt % and aluminum content was intermediate (6 wt % Al), alloy no.2 and 3.



Figure 4-5 Thermal properties of the experimental alloy

However, the melting range was influenced in contrast with the solidus and liquidus point. The melting range was widest when the content of aluminum and nickel were lowest; alloy number 1 (3 wt % Al 0 wt % Ni). The melting range was decreased when the aluminum and nickel content increased. However, the melting range was slightly increased when the nickel content was 6 wt %. The lowest melting ranges of the alloy in this study were found in 9 wt % Al group; alloy number 3, 7, 11, and 14.

Allovgroup	Solidu	ıs temperatu	ıre (°C)	Liquidu	us temperat	ure (°C)	Melting range
Anoygroup	r=1	r=2	average	r=1	r=2	average	(°C)
1	1031.4	1033.1	1032.3	1078.6	1078.5	1078.6	46.3
2	1015.0	1014.4	1014.7	1055.6	1057.3	1056.5	41.8
3	1021.8	1023.1	1022.5	1050.0	1050.2	1050.1	27.7
4	1031.1	1031.8	1031.5	1058.8	1059.4	1059.1	27.7
5	1057.0	1057.8	1057.4	1091.8	1093.2	1092.5	35.1
6	1033.5	1032.0	1032.8	1069.5	1068.0	1068.8	36.0
7	1024.9	1024.7	1024.8	1054.1	1050.4	1052.3	27.5
8	1032.2	1032.0	1032.1	1061.2	1060.7	1061.0	28.9
9	1066.6	1066.1	1066.4	1097.8	1100.2	1099.0	32.7
10	1045.6	1044.8	1045.2	1076.8	1077.5	1077.2	32.0
11	1030.8	1030.8	1030.8	1057.6	1057.7	1057.7	26.9
12	1040.7	1041.1	1040.9	1066.9	1068.6	1067.8	26.9
13	1069.0	1069.4	1069.2	1114.4	1112.3	1113.4	44.2
14	1044.6	1044.7	1044.7	1087.2	1085.1	1086.2	41.5
15	1020.2	1021.3	1020.8	1058.4	1058.1	1058.3	37.5
16	1048.4	1048.7	1048.6	1077.5	1077.7	1077.6	29.1

Table 4-1The results of thermal properties.

4.2 The physical properties and mechanical properties of the experimental Cu-Al-Ni alloy.

According to the difficulty to cast of the alloy number 16 (12 wt %Al, 6 wt % Ni), The tensile and hardness specimen could not be able to completely done. The physical and mechanical property results of this alloy were lacking.

4.2.1 Tensile properties

The mean and standard deviation of the ultimate tensile strength, 0.2% proof stress, modulus of elasticity and elongation after fracture of the experimental alloys demonstrated in Figure 4-6 to 4-9. The different highlight color in the table was showed the statistical difference (p<0.05) and increased as the color indicator illustrated in the figure.

The results of two-way analysis of variance (ANOVA) of the ultimate tensile strength, 0.2% proof stress, modulus of elasticity and elongation after fracture; each and every one are influenced by interaction of aluminum and nickel.

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			Tensile stre	ngth (Mpa)		
	12	4 470.8 (60.2)	8 380.2 (54.3)	12 595.9 (14.2)	16 n/a	maximum
	9	3 436.2 (25.5)	7 504.6 (16.9)	11 505.6 (13.0)	15 539.1 (27.2)	
	6	2 189.4 (13.3)	6 210.0 (14.7)	10 255.2 (25.8)	14 340.9 (37.8)	
Al (Weight %)	3	1 197.3 (11.2)	5 209.8 (14.9)	9 246.8 (21.2)	13 348.0 (44.7)	minimum (p<0.05)
Cu Balan	ice	0 Ni (Weight %)	2	4	6 → (SD in	parentheses]

Figure 4-6 Mean and standard deviation of tensile strength

			0.2 % Proof st	trength (Mpa)		
1	12	4 n/a	8 n/a	12 507.7 (23.8)	16 n/a	maximum
	9	3 168.0 (4.3)	7 180.4 (5.8)	11 198.2 (3.4)	15 246.4 (12.0)	
	6	2 54.5 (3.7)	6 72.2 (2.6)	10 86.3 (9.3)	14 147.0 (10.2)	
Al (Weight %)	3	1 50.7 (3.3)	5 59.1 (5.5)	9 57.8 (4.9)	13 141.6 (10.7)	minimum (p<0.05)
Cu Balan	ce	0 Ni (Weight%)	2	4	\rightarrow (SD in p	arentheses)

Figure 4-7 Mean and standard deviation of 0.2 % proof strength

	Elongation (%)								
		4	8	12	16				
	12	0.1 (0.0)	0.1 (0.1)	1.7 (0.6)	n/a	maximum			
				(<i>)</i>	- *				
		3	7	11	15				
	9	19.5 (1.8)	21.9 (2.1)	23.3 (2.2)	20.4 (2.9)				
		2	6	10	14				
	6	38.4 (5.7)	46.4 (6.6)	47.4 (6.8)	29.9 (3.1)				
I			. ,		. ,				
AI		1	5	9	13				
(Weight %) 3	32.7 (2.5)	42.0 (3.9)	45.1 (4.3)	35.5 (2.9)	minimum			
						(p<0.05)			
		0	2	4	6				
Cu Balance		Ni (Weight %)		•	\rightarrow (SD in	parentheses]			

Figure 4-8 Mean and standard deviation of elongation

The ultimate tensile strength was influenced by both factors without any interaction effect. They increased as the both elements increased. Aluminum showed higher degree of increasing rather than nickel did. The highest ultimate tensile strength was showed on the alloy number 12 (12 wt % Al, 4 wt %Ni). If the alloy number16 could be done, the highest ultimate tensile strength alloy may be demonstrated on this position. (Figure 4-6)

As the same as the ultimate tensile strength, 0.2% proof stress was influenced by both factors and their interaction. They increased as the both elements increased. Aluminum demonstrated higher degree of increasing rather than nickel did. The alloy contained the high aluminum (12 wt %) showed the brittleness of the alloy, as demonstrated extra low elongation on figure 4-8 until the 0.2% proof stress could not be found on alloy number 4 and number 8 (12 wt %Al); Figure 4-7.

As showed in Figure 4-8, the elongation was influenced by both factors and their interaction. They decreased as the aluminum content increased while nickel was counteracting aluminum that means the aluminum make up the alloy more brittleness especially when nickel content was less.



Figure 4-9 Mean and standard deviation of modulus of elasticity

The modulus of elasticity was influenced by both factors and their interaction. They increased when aluminum and nickel content increased , however when aluminum up to 12 wt %, The modulus of elasticity of the alloy were reverse back to lower value due to the drastically increased of the brittleness; low elongation. The higher values were found in the alloy groups of 9 wt % Al and 6 wt % Ni as showed in Figure 4-9.

4.2.2 Surface hardness

The surface hardness was influenced by both factors and their interaction. They increased as the both elements increased. Aluminum demonstrated higher degree of increasing rather than nickel did as the same as the ultimate tensile strength. The highest surface hardness was demonstrated on the alloy number 12; 12 wt % Al, 4 wt % Ni as showed in Figure 4-10.

		Surface har	dness (HV)		
12	4 279.1 (3.0)	8 283.7 (3.0)	12 305.9 (2.5)	16 n/a	maximum
9	3 117.3 (0.9)	7 128.5 (1.6)	11 133.7 (1.1)	15 144.5 (1.1)	
6	2 56.8 (1.2)	6 63.9 (1.3)	10 66.8 (2.8)	14 94.6 (2.0)	
Al (Weight %) 3	1 50.6 (1.6)	5 54.0 (1.7)	9 56.1 (1.0)	13 94.1 (1.1)	minimum (p<0.05)
Cu Balance	0 Ni (Weight %)	2	4	\rightarrow (SD in	parentheses]

Figure 4-10 Mean and standard deviation of the hardness of the experimental alloys

4.3 Study of the corrosion resistance, electrochemical behavior and tarnish resistance of the experimental Cu-Al-Ni alloy.

4.3.1 Electrochemical test

The potentiodynamic pattern of all experimental alloys showed not much difference. Slightly remark on the pattern was found that when the content of aluminum increase, a very smaller reduction in the corrosion current after reached the active peak potential (Ec) was found and demonstrated more stable current density when the potential increased to breakdown potential (Ep); Figure 4-11.

From Table 4-3, the Eocp values showed the thermodynamic of electrocorrosion. The lesser value showed more active to anodic regime. The greater values showed nobler than the lesser. The alloy number 2, 6, 10 (6 wt % Al, 0 to 4 wt % Ni) showed more high value than the others.



Figure 4-11 Polarization curves of 0 wt % Ni alloy with varying Al content

The Ecorr to Ec, Ic, Ip and time to form protection film values demonstrated the alloy passivity. The lesser values showed the better passivity. The Ecorr to Ec, Ic, and time to form film values showed how easy to passivation; lesser is better, while lower Ip values showed higher degree of passivation. The alloy number 10 (Al 6%, Ni 4%) showed all values the smallest demonstrated that this alloy was the most easy passivation and had a highest degree of passivation than the others; Table 4-3, 4-4.

The Ep values showed the stability of the passive film, this value higher is the better. However, alloy number 10 (6 wt % Al, 4 wt % Ni) showed the lowest, this meant that this alloy passive film was the most easiest breakdown and loss their oxide film easily that also demonstrated in the corrosion rate. Alloy number 10 showed the highest corrosion rate than the others.

	Allov	Ip		Allov	Ic		Allov	Ep
	,	(Amps/cm2)		,	(Amps/cm2)		74107	(mV,SCE)
High	8	1.13E-03	Quick	10	1.30E-03	Better	14	175.1
	9	1.20E-03	\uparrow	11	3.20E-03	\uparrow	9	168.3
	7	1.37E-03		3	3.62E-03		4	168.1
	12	1.97E-03		2	4.82E-03		13	137.4
_	6	1.99E-03		15	6.00E-03		7	134.5
ior	1	2.06E-03	uo	8	6.32E-03	3	1	116.5
vat	14	2.10E-03	/ati	9	8.69E-03	e fil	11	116.5
assi	2	2.20E-03	ISSIV	12	1.10E-02	the	15	116.5
ď	13	2.69E-03	ed o	7	1.18E-02	, of	8	114.9
of	3	2.78E-03	y to	14	1.32E-02	lity	3	114.4
gree	5	2.98E-03	oilit	1	1.32E-02	abi	5	108.4
Deg	15	3.20E-03	Ał	4	1.33E-02	St	12	96.0
	11	6.08E-03		6	1.50E-02		2	94.1
	4	6.59E-03		13	1.58E-02		6	84.7
Low	10	7.68E-03	Slow	5	1.78E-02	Worse	10	59.6

Table 4-2	Passivity	of exp	perimen	tal alloys
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Passivity (Corrosion resistance)

 Table 4-3
 Kinetics and Thermodynamics of electrocorrosion of experimental alloys

Killetits	orelectio	CONOSION	mem	louyilai	incs of ef	ectrocorrosion	I		
	Allov	Corr_rate		A		Еоср		ΑΠον	Ecorr
	, aroy	(mmpy)			7.110 y	(mV, SCE)		, arey	(mV, SCE)
High	10	8.77E-04	Pas	sive	3	-214.4	Noble	11	-247.8
	6	5.85E-04	/	\mathbf{h}	6	-218.5		6	-248.2
	1	4.22E-04			11	-222.1		9	-264.8
	5	3.74E-04			9	-227.7		7	-266.9
	13	3.39E-04			7	-236.3		5	-269.8
	2	2.60E-04			5	-247.4		3	-281.6
	3	2.07E-04			2	-247.7		13	-292.6
	9	1.66E-04			10	-247.7		4	-295.4
	4	9.92E-05			4	-256.9		12	-295.4
	15	9.44E-05			8	-266.9		15	-295.4
	11	8.73E-05			13	-267.4		8	-305.9
	8	7.33E-05			15	-277.7		14	-307.6
	14	3.77E-05			12	-292.0		10	-307.7
	12	3.55E-05			1	-292.1		2	-320.0
Low	7	2.44E-05	Act	ive	14	-292.4	Active	1	-333.5

Kinetics of electrocorrosion Thermodynamics of electrocorrosion

Time to form protection film						
	Allov	Time				
	74107	(min)				
Better	4	5.50				
\uparrow	8	6.49				
	5	7.00				
	7	7.06				
	15	7.06				
	12	7.25				
	6	7.28				
	9	7.33				
	13	8.22				
	1	8.37				
	11	8.37				
	3	8.50				
	10	9.04				
	14	9.04				
Worse	2	10.40				

Table 4-4Time to form the oxide protection film

4.3.2 Immersion test

The results of two-way analysis of variance (ANOVA) of color difference (DE*) of the experimental alloys after 0.1 mol/l lactic acid and 0.1 mol/l sodium chloride cyclic immersion test was influenced by both factors and their interaction. All are influenced by interaction of aluminum and nickel.



Figure 4-12 Mean and standard deviation of the color difference

			Lightness chang	ge (CIELAB DL*)		
Ŷ	12	4 7.7 (0.1)	8 15.8 (0.1)	12 13.6 (0.1)	8.0 (0.2)	maximum
	9	3 10.9 (0.1)	7 14.4 (0.1)	11 26.8 (0.1)	15 21.8 (0.1)	
	6	2 34.4 (0.0)	6 34.0 (0.1)	10 35.8 (0.1)	14 33.1 (0.1)	
Al (Weight %)	3	1 43.5 (0.0)	5 36.0 (0.1)	9 42.4 (0.0)	13 37.5 (0.1)	minimum (p<0.05)
Cu Balan	ce	0 Ni (Weight%)	2	4	\rightarrow (SD in p	arentheses)

Figure 4-13 Mean and standard deviation of the lightness change difference

From Figure 4-12 and 4-13 showed the mean and standard deviation of color difference (DE*) and lightness difference (DL*) of the experimental alloys. Both factors were less change when both elements increased. Aluminum showed higher degree of tarnish protection rather than nickel did. The lowest changes were showed on the alloy contained the highest both elements; alloy number 16 (12 wt %Al, 6 wt % Ni). Therefore the entire experimental alloys derived darker than the originating.



Figure 4-14 Mean of the chromaticness a* (Redness) change



Figure 4-15 Mean of the chromaticness b* (Yellowness) change

As the same of colour and lightness difference, in the term of chromaticness CIELAB a* (redness) and CIELAB b*(yellowness), the results demonstrated that the experimental alloys developed paler colour since red on the way to green and yellow towards to blue; Figure 4-14 and 4-15.



Figure 4-16 Elution of copper presented in tarnish test solution

Nevertheless, when checking the elements eluted from the specimen after the immersion test, confirmed that aluminum was acted as the protection elements to prevent the elution of the copper core elements. The content of copper leasing out was totally disappeared, when the aluminum contents increased up to 9 % ; Figure 4-16. This result confirmed that aluminum was acted as the corrosion protection on the alloys. The content of nickel leasing out was increased, at what time the nickel contents increased. The lowest value demonstrated in 6 wt % Al series as showed in Figure 4-17. Fac. of Grad. Studies, Mahidol Univ.



Figure 4-17 Elution of nickel presented in tarnish test



Figure 4-18 Elution of all elements presented in tarnish test solution



Figure 4-19 Mean of all elements presented in tarnish test solution

Even so, in the group of same nickel content all elements leasing out in 9 wt % Al alloy series was lower than others and also alloy number 7 (9 wt % Al 2 wt % Ni) demonstrated the lowest value as showed in Figure 4-18 and 4-19.

4.4 Study of the bio-compatibility properties of the experimental Cu-Al-Ni alloy.

From Table 4-5 and figure 4-20 showed the results of cytotoxic to the cell of the experimental alloys. All alloys showed the moderate cytotoxicity to the cell as the normal alloys (positive control) used in dentistry do. However, the higher content of nickel showed more decolorization zone. The highest content of nickel showed the biggest decolorization zone that cause the decolorization index up to level 3. This confirmed the negative effect of the nickel to the human biocompatibility.

Specimen	Decolorization zone	Decolorization index	Lysis index	Cell response	Cytotoxic interpretation
	(mm)				
Control (-)	0	0	0	0/0	non
Control (+)	5.5	3	2	3/2	moderately
1	3.4	2	2	2/2	moderately
2	3.4	2	2	2/2	moderately
3	3.9	2	2	2/2	moderately
4	3.8	2	2	2/2	moderately
5	4.0	2	2	2/2	moderately
6	4.0	2	2	2/2	moderately
7	3.9	2	2	2/2	moderately
8	4.3	2	2	2/2	moderately
9	4.5	2	2	2/2	moderately
10	4.0	2	2	2/2	moderately
11	4.5	2	2	2/2	moderately
12	4.5	2	2	2/2	moderately
13	5.9	3	2	3/2	moderately
14	5.0	3	2	3/2	moderately
15	5.3	3	2	3/2	moderately
16	5.8	3	2	3/2	moderately

Table 4-5The results of cytotoxicity test

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	Cell response (lysis index/decolorization zone)										
\uparrow	12	4 2/2	8 2/2	12 2/2	16 3/2						
	9	3 2/2	7 2/2	11 2/2	15 3/2	control - 0/0					
	6	2 2/2	6 2/2	10 2/2	14 3/2	control + 3/2					
Al (Weight %)	3	1 2/2	5 2/2	9 2/2	13 3/2						
Cu Balan	ice	0 Ni (Weight %) -	2	4	6 →						

Figure 4-20 Cytotoxic interpretation of the biocompatibility
CHAPTER V DISCUSSION

The technique mostly used to fabricate post and core is the "*cast post and core*". Custom cast posts and cores may be fabricated from a variety of materials and generally are prepared as single units. They reproduce the contours of the prepared canal and adapt well to the canal morphology and are commonly chosen in those situations where the canal is irregularly shaped.^{13, 22, 30, 31} Favorable internal adaptation of the post will distribute the internal stresses relatively uniformly without stress concentration.³ The cast post and core will often provide greater resistance to rotation than a prefabricated post due to the excellent internal adaptation and the irregularity in shape.

An important requirement for post materials is stiffness. The stiffness of the post (a combination of cross-sectional geometry and modulus of elasticity) is an extremely important characteristic. Insufficient stiffness of the post permits micromovement of the core and distortion of the restoration at the margins during function or cement breakdown and recurrent caries. However, the post must not be so stiff that it cannot deform compatibly with the root as it bends under functional loads. Failure to do so, root fracture could be the consequence. The material selected for post fabrication should also have a modulus of elasticity close to the root.

Kinny J.H. ¹⁵⁴ reviewed the modulus of elasticity of the root dentin from the experiment articles and found that the modulus of elasticity of the root dentin values are ranged from 10 to 30 GPa with the average and standard deviation for 13.2 ± 4.0 GPa. However, the available alloy in dentistry has much higher modulus of elasticity value than the root dentin such as gold alloys are around 70 to 100 GPa, Palladium alloys are around 120 GPa, Silver alloys are 100 GPa and base metal alloy (Ni-Cr, Co-Cr) are 160 to 250 GPa while titanium are 100 to 110 GPa.¹⁵⁵

Therefore a desirable value of elastic modulus of experimental alloy should close to root dentine or at lease comparable to gold alloy (70-100 GPa). In addition to ISO standard, the other properties of experimental alloy should conform or pass the standard. As stated by the international standard. ISO 22674:2006; Dentistry - Metallic materials for fixed and removable restorations and appliances: Type 2 intended for single tooth fixed restorations, the minimum values of 0.2 % proof strength and percentage elongation after fracture are 180 MPa and 10 %, respectively.

5.1 The physical and mechanical properties

The experimental alloy number 16 (12 wt % Al, 6 wt % Ni) in this study could not be cast completely for tensile specimens. The casting found a lot of defects and incomplete.

The Solidus and liquidus temperature of all 16 experimental alloys are not much different. They ranged from 1014.7 to 1069.2 °C for solidus point and 1050.1 to 1113.4 °C for liquidus point. They slightly decreased as the aluminum content increased and increased as the nickel content increased. However, the melting range was only influenced by the aluminum content. They decreased as the aluminum content increased. This was conformed to the Cu-Al-Ni ternary phase diagram which showed in Figure 5-1.¹⁶⁴

This diagram is a vertical section at temperature 400-1200 °C and partial composition of 62.3-93.6 wt % Cu, 0-2 wt % Al and 5.3-6.4 wt % Ni. The melting range was shown nearly the same small range throughout the diagram with solidus and liquidus temperature around 1000-1100 °C. This melting range is close to commercial available gold alloy which have a great benefit for ease casting, high castability and low fabrication cost.



Alexander W.O.: "COPPER-RICH NICKEL-ALUMINIUM-COPPER-ALLOYS. , J. Inst. Met. 63 (1938) 163–189. Figure 5-1. Cu-Al-Ni ternary phase diagram

This diagram is a vertical section at temperature 400-1200 °C and partial composition of 62.3-93.6 wt % Cu, 0-2 wt % Al and 5.3-6.4 wt % Ni. The melting range was shown nearly the same small range throughout the diagram with solidus and liquidus temperature around 1000-1100 °C. This melting range is close to commercial available gold alloy which have a great benefit for ease casting, high castability and low fabrication cost.

From the tensile properties, both nickel and aluminum contents were influenced the tensile properties of the experimental alloys. The tensile strength, proof strength gradually increased as the aluminum contents increased. The proof stress could not be found while the elongation was gradually decreased until showed less than 1 wt % when the aluminum content increased to 12 wt %. This meant that the increasing of aluminum content caused the brittleness of the experimental copper alloy. The experimental alloy with 12% aluminum content may not be suitable for using in this aspect due to their high brittleness until presented no proof stress, less elongation and could not be casted. The elastic modulus was also conformed to the changing of the strength and elongation. They found increasing when the strength gradually increased while the elongation slightly decreased leading to the reversing down in the value of the elastic modulus. This may be due to the form phase of

aluminium with copper, the alloy will form the single-phase (face-centered cubic) alpha alloys if the alloy containing aluminium less than 8%. This alloy has a high ductility and suitable for cold working such as wiring, tubing and sheeting. If the aluminium content is increased to between 8% and 10%, the alloys are highly strengthening by appearance of the harder body-centered cubic β phase. This phase make the alloy strengthen and more suitable for casting. However, if the aluminium content is increased over 10%, the alloys are progressively greater strength and hardness but difficult to cast as the nature of copper alloy: low castability.¹⁵⁶ The nickel content exhibited the influenced only on tensile strength and proof stress and slightly on surface hardness while it exhibited no influenced on the elongation. The tensile strength and proof strength increased by the way of the nickel content increased, leading to increase of the elastic modulus. This meant the nickel increased the strength of the copper alloy without increase the brittleness of the alloy. From the alloy phase diagram, nickel can form complete solid solution to copper to be singlephase alpha alloy throughout the diagram (Figure.5-2). This homogeneous phase alloy make the alloy strengthen without brittle.



V. Raghavan. Phase Diagram Evaluations: Section II. Journal of Phase Equilibria and Diffusion (2006); 27:390

Figure 5-2. Cu-Ni phase diagram

As commend in the above that a desirable value of elastic modulus of experimental alloy should close to root dentine or at lease comparable to gold alloy (70-100 GPa). and the other properties should pass the ISO 22674:2006; Dentistry -

Metallic materials for fixed and removable restorations and appliances: Type 2 intended for single tooth fixed restorations, the minimum values of 0.2 % proof strength and percent elongation after fracture are 180 MPa and 10 %, respectively. Only the experimental alloy no 11,12,15 (contain Al \geq 9 wt. % with Ni \geq 4 wt. %) can passed the minimum value of 0.2% proof stress, while all alloy which contained Al \leq 9 wt. % can passed the minimum value for percent elongation. All alloy showed the elastic modulus around 60-140 GPa which close to commercial gold and palladium alloy.

In conclusion to mechanical properties, only alloy no 11 and 15 (9 wt. % Al, 4-6 wt. % Ni) can pass the ISO specification for good alloy using for post and core application and showed elastic modulus 122.8 and 113.7 GPa respectively.

5.2 The corrosion

The electrochemical behavior of the experimental alloys was shown not much difference in the potentiodynamic pattern. More aluminum content showed more stable of passive film. The nickel contents showed no any significant from each others. This was conformed to the color difference result that aluminum plays an active role on the corrosion protective of this alloy. Additionally, formation of very thin protective film may be due to characters of these alloys and using 0.9% NaCl as test media. Comparing to potentiodynamic curve of copper in 3.5% NaCl,¹⁶⁰ they look like in the same pattern: Figure 5-3. The corrosion reaction of copper, especially in a solution containing chloride ions, were quite complicated, but the reactions might briefly be described as the formation of copper (I) (cuprous) and copper (II) (cupric) oxides, together with insoluble hydrated chlorides. Mayer and Nally ¹⁶¹ indicated that 0.9% saline solution was considered to be more aggressive than saliva and its artificial substitutes, because the chloride which was six time higher. Moreover, it lacks of some component which might show a corrosion inhibited action and a buffer capacity such as phosphate. Marek and Topfl¹⁶² suggested that 1% NaCl was unsuitable for measurement other than the screening test for generalized corrosion.



Figure 5-3 Potentiodynamic scan for copper in 3.5% NaCl solution

One of the problems associated with the corrosion studies of copper alloys is that knowledge of the electrochemical environment of the oral cavity is limited. This makes the preparation of testing condition and the interpretation of in vitro data somewhat hypothetical.

The potential occurred in the oral cavity has been reported by Ewers and Greener ¹⁶³, who produced and envelop of electrochemical activity for the oral cavity by collecting data relating to the oxidation potential and pH. They reported the oxidation potential ranged from -58 to +212 mV, SCE and the pH ranged from 6.1 to 7.9. Regard to the scan conditions in this study, the potential was increased up to 1000mV. The final potential was much higher than the potential occurred in the oral cavity. So, we could not study the corrosion behavior of these alloys under conditions that can occur in the oral cavity. Such a high potential and corrosive environment caused the copper alloys corroded aggressively. However, this situation might not have occurred in the oral cavity. Therefore, if someone needs to correlate the corrosion behavior of in vitro study with clinical aspects, he should limit the potential within the potential range of the oral cavity (-58 to +212 mV).

In conclusion, the electrochemical behaviors of all experimental alloys are not much difference. All alloys can withstand in the limited oxidation potential ranged of the oral cavity (-58 to +212 mV) due to the passivity potential (Ep) was stable within the oral oxidation potential range.

5.3 The biocompatibility, tarnish and elements elution

The goal of all biocompatibility testing is a screening test for dental materials by simulates conditions that would exist in the oral environment. Biocompatibility Standard ISO 7405: 2008¹⁶⁸ has suggested a series of tests to ascertain that a medical or dental materials and devices is compatible. This aspect would also related to the corrosion behavior of the materials especially elements release in the simulate environment.

The result of biocompatibility test presented the moderately cytotoxicity comparable to the positive control, but lesser in term of cell response: 3/2 and 2/2respectively. However, the higher content of nickel exhibited more decolorization zone. The highest content of nickel in this study: 6 wt % revealed the biggest decolorization zone that cause the decolorization index up to "level 3" equally a positive control. This can be implying that nickel cause a negative effect and toxic to the cell. The nickel ion was found to induce the cell (human cell lines) to release the intracellular enzyme lactic dehydrogenase which stimulated the lactic acid production. Lactic acid in the cell causes DNA damage leading to response of the cell to repair DNA, cell cycle arrest or apoptosis.¹⁵⁷ Several studies tried to show the evidence of the negative effect of nickel to human, numerous nickel compounds are believed to be carcinogens. However, the most evidence found on nickel contained appliances is contact allergy. The weighted average prevalence of the allergy which collected from the data from 1966-2007 was 19.5% of the population in all ages and all country. The entire evidence came from jewelry pieces and household items. This is trusted to be due to a galvanic reaction.¹⁵⁸ More interesting things is reactions to dental and orthodontics hardware appliances are rare.¹⁵⁹

The results of immersion test by means of color difference (DE*), lightness difference (DL*), chromaticness CIELAB a* (redness) and CIELAB b*(yellowness) including the elements release found that the effect was depending upon the aluminum content that the higher content of aluminum, the less of color change. This is due to the aluminum oxide (Al₂O₃) corrosion protective film on the surface. The amounts of the oxide on the surface depend upon the amount of aluminum containing in the alloys.

However, the element release results showed the less release on the series of alloys containing 2 wt % Ni. This would be explained by the reaction of the nickel to aluminum. Small amount of nickel will be able to form the complete solid solution to aluminum and the rest of nickel is free in the alloy.

In conclusion, the entire experimental alloy are changed in color when immersion to paler in color and darker in lightness. The degree of change was less when the Al and Ni content were increased. This was conformed to the amount of element release; the amount was less when the Al and Ni content were increased.

While the biocompatibility showed moderate level 2 cytotoxic in majority of experimental alloy except the alloy contained Ni 6%, which showed moderate level 3 cytotoxic equally to positive control.

CHAPTER VI CONCLUSION

As stated by the international standard: ISO 22674:2006; Dentistry -Metallic materials for fixed and removable restorations and appliances; Type 2 intended for single tooth fixed restorations, the minimum values of 0.2 % proof strength and percentage elongation after fracture are 180 MPa and 10 %, respectively. (Table 7.1) The percentage elongation of experimental alloys excluding group of 12 wt % Al in this study were higher than 20 %. However, only alloys in group of 9 wt % Al were complied with the standard of 0.2 % proof strength.

	Proof strength of 0.2 %	Elongation after fracture	Young's modulus
Туре	non-proportional extension		
	MPa	%	GPa
	minimum	minimum	minimum
0	-	-	-
1	80	18	-
2	180	10	-
3	270	5	-
4	360	2	-
5	500	2	150

 Table 7.1
 ISO standard: Statement of mechanical properties

ISO specification did not specified the minimum value of tensile strength and modulus of elasticity intended for this type of alloy, but these properties of the alloys for post and core materials should have no less than that of dental gold alloys; around 420 MPa and 70 to 100 GPa respectively.¹¹³ Therefore, the moduli of elasticity $(63.9\pm5.1$ to 139.1 ± 14.2 GPa) of the entire experiment alloys were on the point of dental gold alloy while these values were higher than that of carbon fiber or glass fiber posts (14 to 18 GPa).⁹³ Though some researchers^{31, 93} revealed that occlusal loads possibly will cause the carbon fiber post generated flexible condition, which activated micro-movement of the core. In this situation the dental restorations possibly were unsuccessful in a short time. On the other hand, these alloys were much cheaper. In this aspect, this significance was promising to be developed in further studies.

The potentiodynamic polarization technique is a fundamental and widely used for corrosion testing *in vitro*. Therefore, the entire potentiodynamic curves of experimental alloys had a long passive region. It was assumed that nickel was in a transitional element group as copper, thus it arranged into solid solution and generated face cubic center phase with copper once small amounts were added.¹⁶⁵

In this study, we did not quantitatively analyze the change of alloy composition in entire specimens and electrolyte after the corrosion resistance test. Hence, we could not identify the structures of oxides and chlorides compounds, passive film, or corrosion products. Further studies will be needed to identify the film of these alloys. It should be noted that the formation of oxides and chlorides, and corrosion products of these alloys also affected their microstructures that might degrade some mechanical properties.

The results of immersion test by means of color difference (DE*), lightness difference (DL*), chromaticness CIELAB a* (redness) and CIELAB b* (yellowness) presented that the entire properties developed paler and effect was depending upon both aluminum and nickel content but aluminium related more significant. Nevertheless, those factors were less change when both elements increased (9 to 12 wt % Al and 2 to 6 wt % Ni alloy series).

The result of biocompatibility presented the highest content of nickel in this study; 6 wt % Ni, revealed the largest cell response equally to the positive control. For this reason these alloy series were not suitable in this situation.

The elements elution presented the less release on the series of alloys containing 2 wt % Ni. This would be explained by the reaction of the nickel to aluminum. Small amount of nickel will be able to form the complete solid solution to aluminum and the rest of nickel is free in the alloy. Though, the total metal ion release from the experiment alloys into the test solution (20.3 to 143.9 μ g/cm²/7d) existed not

greater than the maximum value of the ISO standard statement. (Shall not exceed 200 $\mu g/cm^2/7d$).¹⁶⁶

Regarding, the entire properties of 9 wt % Al containing 2 and 4 wt % Ni alloy series seemed to be the most suitable for post and core material since they had a higher, better and quicker in corrosion resistance behaviors but then lower in corrosion rate than the others. Along with, moderately toxicity that cell response presented better than positive controlled specimen together with reasonable strength.

For those reasons, these alloy series have a potential to present for dental post and core application and capable for further development as appropriate post and core materials.

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APPENDICES

APPENDIX A

Experimental alloy compositions

Alloy group		Contents		
Alloy Broop	Ni (weight %)	Al (weight %)	Cu (weight %)	
1	-	3	97	
2	-	6	94	
3	-	9	91	
4	-	12	88	
5	2	3	95	
6	2	6	92	
7	2	9	89	
8	2	12	86	
9	4	3	93	
10	4	6	90	
11	4	9	87	
12	4	12	84	
13	6	3	91	
14	6	6	88	
15	6	9	85	
16	6	12	82	

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Thermal properties

Allov	Soldus	Soldus (° C)		us (°C)	Meltingr	Melting range (°C)		
Аноу	mean	SD	mean	SD	mean	SD		
1	1032.3	1.0	1078.6	0.1	46.3	1.0		
2	1014.7	0.3	1056.5	1.0	41.7	1.3		
3	1022.5	0.8	1050.1	0.1	27.7	0.6		
4	1031.5	0.4	1059.1	0.3	27.7	0.1		
5	1057.4	0.5	1092.5	0.8	35.1	0.3		
6	1032.8	0.9	1068.8	0.9	36.0	0.0		
7	1024.8	0.1	1052.3	2.1	27.4	2.0		
8	1032.1	0.1	1061.0	0.3	28.9	0.2		
9	1066.4	0.3	1099.0	1.4	32.7	1.7		
10	1045.2	0.5	1077.2	0.4	32.0	0.9		
11	1030.8	0.0	1057.7	0.1	26.9	0.1		
12	1040.9	0.2	1067.8	1.0	26.9	0.8		
13	1069.2	0.2	1113.4	1.2	44.2	1.4		
14	1044.7	0.1	1086.2	1.2	41.5	1.3		
15	1020.8	0.6	1058.3	0.2	37.5	0.8		
16	1048.6	0.2	1077.6	0.1	29.1	0.1		



Thermal properties chart





APPENDIX B MECHANICAL PROPERTIES

Mechanical properties: Table

Tensile	Мра									
Allow	specimen									
Anoy	1	2	3	4	5	Average	S.D.			
1	195.1	214.2	202.0	189.0	186.1	197.3	11.2			
2	176.4	202.3	180.8	205.2	182.2	189.4	13.3			
3	425.2	464.9	399.2	452.6	439.1	436.2	25.5			
4	456.1	509.2	546.4	388.4	454.0	470.8	60.2			
5	193.2	206.5	220.6	229.3	199.6	209.8	14.9			
6	193.8	212.6	230.1	215.9	197.5	210.0	14.7			
7	494.3	496.8	533.0	507.0	492.0	504.6	16.9			
8	407.4	444.8	305.3	395.1	348.3	380.2	54.3			
9	267.2	263.0	215.1	251.7	237.0	246.8	21.2			
10	278.8	252.5	273.3	258.1	213.3	255.2	25.8			
11	493.3	516.8	521.2	503.8	493.2	505.6	13.0			
12	607.0	608.6	594.3	596.6	573.1	595.9	14.2			
13	392.9	317.3	288.0	357.3	384.5	348.0	44.7			
14	280.4	335.7	376.3	368.6	343.7	340.9	37.8			
15	578.9	542.0	545.2	506.3	523.2	539.1	27.2			

Elongation	%							
Allow	specimen							
Alloy	1	2	3	4	5	Average	S.D.	
1	33.0	30.4	34.9	35.4	29.8	32.7	2.5	
2	33.3	41.2	42.5	43.7	31.3	38.4	5.7	
3	17.9	20.4	17.3	20.6	21.2	19.4	1.8	
4	0.1	0.1	0.1	0.1	0.1	0.1	0.0	
5	41.7	47.2	37.3	39.6	44.4	42.0	3.9	
6	55.5	42.3	50.2	45.6	38.6	46.4	6.6	
7	20.5	22.0	25.0	19.4	22.7	21.9	2.1	
8	0.1	0.1	0.0	0.0	0.1	0.1	0.1	
9	43.1	45.9	41.6	42.6	52.1	45.1	4.2	
10	46.6	38.2	50.4	56.7	45.1	47.4	6.8	
11	20.5	25.4	23.6	25.2	21.6	23.3	2.2	
12	1.9	1.0	2.1	2.3	1.2	1.7	0.6	
13	39.8	36.5	34.8	32.1	34.4	35.5	2.9	
14	32.8	35.9	38.3	35.2	23.1	33.1	5.9	
15	21.6	20.6	15.8	20.4	23.7	20.4	2.9	

Mechanical properties: Table (cont.)

0.2% Proof stress	MPa

Allov		S	pecimen				
Alloy	1	2	3	4	5	Average	S.D.
1	53.2	54.5	49.1	50.4	46.3	50.7	3.3
2	57.6	55.5	51.1	50.1	58.1	54.5	3.7
3	171.0	162.0	164.9	171.0	171.2	168.0	4.3
4	-	-	-	-	-	-	-
5	53.3	64.3	62.4	62.2	53.1	59.1	5.4
6	69.6	74.0	69.6	75.2	72.4	72.2	2.6
7	160.0	163.9	164.7	150.6	162.9	160.4	5.8
8	-	-	-	-	-	-	-
9	52.6	55.5	57.8	65.7	57.3	57.8	4.9
10	78.8	97.7	93.3	76.0	85.7	86.3	9.2
11	204.0	197.0	196.5	195.2	198.3	198.2	3.4
12	534.0	519.9	502.7	470.6	511.1	507.6	23.7
13	155.1	127.8	137.1	138.6	149.3	141.6	10.7
14	143.1	160.2	148.3	151.0	132.6	147.0	10.2
15	260.4	234.2	258.0	239.2	240.1	246.4	12.0

Modulus	GPa								
Allow	specimen								
Alloy	1	2	3	4	5	Average	S.D.		
1	74.7	63.5	62.8	64.9	70.4	67.2	5.1		
2	57.5	71.7	67.7	68.2	67.7	66.6	5.3		
3	111.2	115.6	115.5	140.0	130.0	122.5	12.1		
4	80.0	100.2	97.1	106.6	88.5	94.5	10.4		
5	91.8	89.9	109.1	108.3	92.7	98.3	9.5		
6	87.5	104.4	109.8	91.9	93.3	97.4	9.3		
7	99.3	100.7	115.9	95.9	107.9	103.9	8.0		
8	84.0	53.2	76.9	121.7	70.5	81.3	25.3		
9	120.4	94.9	109.8	115.9	100.1	108.2	10.7		
10	111.3	105.1	108.3	102.3	122.8	110.0	7.9		
11	133.5	135.5	120.4	110.9	113.6	122.8	11.3		
12	70.1	63.9	60.5	60.1	64.7	63.9	4.0		
13	147.1	80.1	132.5	112.2	126.6	119.7	25.4		
14	134.7	138.1	162.6	124.4	135.4	139.1	14.2		
15	119.3	117.3	127.0	95.4	109.6	113.7	12.0		

Mechanical properties: Table (cont.)

Hardness	HV						
		S	pecimen				
Anoy	1	2	3	4	5	Average	S.D.
1	50.1	52.1	52.5	49.6	48.7	50.6	1.6
2	55.3	56.8	58.0	56.0	57.8	56.8	1.2
3	116.8	116.0	118.4	117.8	117.5	117.3	0.9
4	281.5	282.1	279.0	278.4	274.5	279.1	3.0
5	53.7	56.3	53.8	51.6	54.8	54.0	1.7
6	64.6	62.9	63.8	65.8	62.4	63.9	1.3
7	129.2	130.4	129.4	127.2	126.5	128.5	1.6
8	282.2	280.9	284.0	288.7	282.8	283.7	3.0
9	55.8	54.9	55.6	56.7	57.5	56.1	1.0
10	65.8	71.5	65.8	64.4	66.5	66.8	2.7
11	132.5	133.7	135.4	133.5	133.3	133.7	1.1
12	307.0	304.6	309.0	306.7	302.4	305.9	2.5
13	95.3	94.8	92.9	94.6	93.0	94.1	1.1
14	94.1	94.9	92.7	97.9	93.7	94.6	2.0
15	144.8	146.1	144.5	143.7	143.2	144.5	1.1

Mechanical properties: Chart







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Mechanical properties: Chart (cont.)





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APPENDIX C ELECTROCHEMICAL TEST

Potentiodynamic polarization curves



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Potentiodynamic polarization curves (cont.)



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Potentiodynamic polarization curves (cont.)


















3 wt % Al Group





6 wt % Al Group

9 wt % Al Group



Potentiodynamic polarization curves (cont.)

12 wt % Al Group



0 wt % Ni Group



Potentiodynamic polarization curves (cont.)

2 wt % Ni Group



4 wt % Ni Group



Potentiodynamic polarization curves (cont.)

6 wt % Ni Group



Tafel fit table

Tafel fit	t results						
Alloy	Ва	Вс	lo	Eo	Еоср	Ecorr	lcorr
	(mV)	(mV)	(Amps/cm2)	(mV)	(mV)	(mV)	(Amps/cm2)
1	90.0	79.6	2.8E-07	-295.19	-247.7	-295.35	2.4E-09
2	55.3	79.7	1.4E-07	-233.47	-214.4	-333.54	5.0E-09
3	119.1	127.9	4.1E-07	-320.52	-302.6	-319.95	1.3E-08
4	44.6	97.6	8.9E-08	-278.34	-266.9	-281.58	6.4E-09
5	64.5	72.6	4.7E-07	-270.41	-247.4	-269.75	2.1E-08
6	65.7	69.3	2.8E-07	-249.67	-218.5	-248.19	1.8E-08
7	99.1	86.9	9.6E-07	-274.57	-236.3	-266.9	3.4E-07
8	95.5	102.9	3.2E-07	-306.2	-292.1	-305.9	4.8E-09
9	63.9	59.8	1.3E-07	-261.86	-227.7	-264.83	9.6E-09
10	101.9	162.0	3.8E-07	-249.63	-222.1	-247.83	5.4E-08
11	83.9	87.0	2.8E-07	-308.09	-256.9	-307.67	5.6E-09
12	84.1	74.5	2.4E-07	-295.19	-292	-295.35	2.4E-09
13	80.6	166.9	2.0E-07	-294.51	-267.4	-292.59	2.0E-08
14	89.7	79.4	2.8E-07	-295.19	-292.4	-295.35	2.4E-09
15	94.4	137.6	2.5E-07	-308.07	-277.7	-307.64	6.1E-09

Potentiodynamics chart







Potentiodynamics result chart (cont.)







Narration of Corrosion rate

Elements	Atomic Weight (g/mole)	Possible Valence
Cu	63.546	1, 2
Al	26.982	3
Ni	58.700	2, 3

Equivalent Weight (g/equivalent)

		Cu Valence	e	
Allow	Low	vest	Seco	ond
Alloy	Cu	=1	Cu	=2
1	53.8	-	29.5	-
2	46.6	-	27.6	-
3	41.1	-	25.9	-
4	36.8	-	24.4	-
		Ni Valence	9	
	Cu	=1	Cu	=2
Alloy	Lowest	Second	Lowest	Second
	Ni=2	Ni=3	Ni=2	Ni=3
5	52.7	51.8	29.5	29.2
6	45.8	45.1	27.5	27.3
7	40.5	40.0	25.8	25.6
8	36.3	35.9	24.3	24.1
9	51.7	50.0	29.4	28.9
10	45.1	43.7	27.5	27.0
11	39.9	38.9	25.8	25.4
12	35.8	35.0	24.3	24.9
13	50.8	48.3	29.4	28.5
14	44.3	42.4	27.5	26.7
15	39.3	37.8	25.8	25.1

Narration of Corrosion rate (cont.)

	CR	lcor	r*K*EW
	en -		dA
Where:			
	CR	=	The corrosion rate. Its units are given by the choice of K
	lcorr	=	The corrosion current in amps
	К	=	A constant that defines the units for the corrosion rate (mmpy=3272)
	EW	=	The equivalent weight in grams/equivalent
	d	=	Density in grams/cm ³
	А	=	Sample area in cm ²

Corrosion rate

Corrosion ra	te									
	Density		E a colorada				Corr	osion rate (m	mpy)	
АПОУ	(g/cm3)		Equivare	nt weight						Average
1	7.6	53.8	-	29.5	-	1.10E-03	-	6.73E-04	-	8.88E-04
2	7.2	46.6	-	27.6	-	7.14E-04	-	4.55E-04	-	5.85E-04
3	6.8	41.1	-	25.9	-	2.54E-04	-	1.60E-04	-	2.07E-04
4	6.5	36.8	-	24.4	-	1.19E-04	-	7.91E-05	-	9.92E-05
5	7.6	52.7	51.8	29.5	29.2	4.83E-04	4.75E-04	2.70E-04	2.68E-04	3.74E-04
6	7.2	45.8	45.1	27.5	27.3	4.38E-04	4.17E-04	2.54E-04	2.46E-04	3.39E-04
7	6.8	40.5	40.0	25.8	25.6	1.07E-04	1.04E-04	6.94E-05	6.82E-05	8.73E-05
8	6.5	36.3	35.9	24.3	24.1	8.82E-05	8.71E-05	5.91E-05	5.86E-05	7.33E-05
9	7.6	51.7	50.0	29.4	28.9	5.57E-04	5.55E-04	2.88E-04	2.89E-04	4.22E-04
10	7.2	45.1	43.7	27.5	27.0	3.26E-04	3.23E-04	1.95E-04	1.95E-04	2.60E-04
11	6.8	39.9	38.9	25.8	25.4	2.14E-04	2.07E-04	1.22E-04	1.20E-04	1.66E-04
12	6.5	35.8	35.0	24.3	24.9	4.24E-05	4.14E-05	2.88E-05	2.95E-05	3.55E-05
13	7.6	50.8	48 3	29.4	28 5	1 16F-04	1 12F-04	7 60F-05	7 41F-05	9 44F-05
14	7.2	44 3	42.4	27.5	26.7	4 74F-05	4 54E-05	2 94E-05	2 86E-05	3 77E-05
15	6.8	39.3	37.8	25.8	25.1	3.06E-05	3.01E-05	1.84E-05	1.82E-05	2.44E-05

APPENDIX D CYCLIC IMMERSION TEST

CIE 014-4.3/E:2007, Colorimetry- Part 4: <u>CIE 1976 L* a* b*</u> colour space, CIE Central Bureau, Vienna, Austria.



CIE 1976 L* a* b* colour space = CIELAB

Ligh	tness
------	-------

L* ΔL* ΔL*, (DL*) =	CIE CIE L1'	LAB lightness LAB lightness difference * - LO*	
Chromaticness			
a*, b*	CIE	LAB a*, b* coordinates	
∆a*, (Da*)	CIE	LAB a* chromaticness difference	$= a_1^* - a_0^*$
Δb*, (Db*)	CIE	LAB b* chromaticness difference	$= b_1^* - b_0^*$
Colour			
ΔΕ* <i>,</i> (DE*)	CIE	LAB colour difference	
$\Delta E^* = \sqrt{(\Delta L^*)^2} +$	+(∆a*) ² +	$\overline{\left(\Delta b^{*}\right)^{2}}$	

	DI	*	Da	*	Dł	0*	D	E
Alloy	mean	SD	mean	SD	mean	SD	mean	SD
1	43.5	0.03	13.1	0.14	24.1	0.27	51.5	0.1
2	34.4	0.04	5.7	0.14	24.3	0.22	42.5	0.1
3	10.9	0.10	6.7	0.65	2.8	0.18	13.1	0.3
4	7.7	0.13	5.2	0.58	8.0	0.26	12.3	0.3
5	36.0	0.05	6.6	0.09	15.0	0.14	39.5	0.1
6	34.0	0.05	4.1	0.10	17.0	0.14	38.2	0.1
7	14.4	0.09	5.8	0.26	6.2	0.23	16.7	0.1
8	15.8	0.07	5.0	0.28	3.3	0.34	16.9	0.1
9	42.4	0.05	5.9	0.13	13.1	0.17	44.7	0.1
10	35.8	0.09	4.0	0.30	14.3	0.17	38.8	0.1
11	26.8	0.05	3.7	0.21	15.1	0.20	31.0	0.1
12	13.6	0.11	4.2	0.35	3.5	0.28	14.7	0.2
13	37.5	0.10	5.2	0.15	9.8	0.19	39.1	0.1
14	33.1	0.10	4.0	0.22	11.3	0.22	35.2	0.2
15	21.8	0.08	2.2	0.30	7.2	0.19	23.1	0.1
16	8.0	0.17	2.5	0.39	1.4	0.36	8.5	0.2

Lightness, Chromaticness and Colour difference table

Colour difference chart





Lightness difference chart





Chromaticness difference chart

Redness



Yellowness



Elution of elements table

Elution of elements in the test solution							
Allov	μg/cm²/7d						
Аноу	Cu	Al	Ni	Total			
1	110.1	6.4	0.2	233.3			
2	127.7	16.0	0.2	287.8			
3	0.2	61.9	0.2	124.6			
4	0.1	81.7	0.2	164.1			
5	97.3	2.8	9.0	218.0			
6	31.2	11.2	1.4	87.6			
7	0.4	14.7	5.1	40.6			
8	0.0	42.7	7.4	100.1			
9	109.5	6.7	7.9	250.2			
10	93.2	6.8	5.4	211.7			
11	0.9	24.6	8.8	163.2			
12	0.3	38.4	11.5	102.8			
13	90.7	5.4	9.8	209.9			
14	35.0	3.4	6.1	88.4			
15	0.4	22.8	13.3	68.4			
16	0.1	59.5	13.9	144.7			

Elution of elements chart



Elution of elements chart (cont.)







APPENDIX E BIOCOMPATIBILITY

Narration of decolorization index

Decolorization	Description			
index				
0	No detectable decolorization zone around or under specimen			
1	Decolorization zone limited to area under specimen			
2	Decolorization zone extends less than 0.5 cm beyond specimen.			
3	Decolorization zone extends 0.5 cm to 1.0 cm beyond specimen.			
4	Decolorization zone extends greater than 1.0 cm beyond specimen			
	but does not involve entire dish.			
5	Decolorization zone involves entire dish.			

Narration of lysis index

Lysis index	Description
0	No observable cytotoxicity.
1	Less than 20% of the decolorized zone affected.
2	20% to < 40% of the decolorized zone affected.
3	40% to < 60% of the decolorized zone affected.
4	60% to < 80% of the decolorized zone affected.
5	Greater than 80% of the decolorized zone affected.

Narration of cell response and interpretation

_					
_	Scale	Cell Response	Interpretation		
-					
	0	0/0	Non cytotoxic		
	1	1/1	Mildly cytotoxic		
	2	2/2 to 3/3	Moderately cytotoxic		
	3	4/4 to 5/5	Severely cytotoxic		
-					

Cytotoxic interpretation

Specimen	Decolorization zone (mm)	Decolorization index	Lysys index	Cell response	Cytotoxic interpretation
Control (-)	0	0	0	0/0	non
Positive (+)	5.5	3	2	3/2	moderately
1	3.4	2	2	2/2	moderately
2	3.4	2	2	2/2	moderately
3	3.9	2	2	2/2	moderately
4	3.8	2	2	2/2	moderately
5	4.0	2	2	2/2	moderately
6	4.0	2	2	2/2	moderately
7	3.9	2	2	2/2	moderately
8	4.3	2	2	2/2	moderately
9	4.5	2	2	2/2	moderately
10	4.0	2	2	2/2	moderately
11	4.5	2	2	2/2	moderately
12	4.5	2	2	2/2	moderately
13	5.9	3	2	3/2	moderately
14	5.0	3	2	3/2	moderately
15	5.3	3	2	3/2	moderately
16	5.8	3	2	3/2	moderately

Decolorization index



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 1



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 2

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Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 3



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 4



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 5



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 6

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Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 7



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 8



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 9



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 10

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Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 11



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 12



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 13



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 14

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Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 15



Upper Left: PVC (+ve) Upper Right: Negative (-ve) Lower Left and Right: material 16

Lysis index



Negative Control



Positive Control

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Alloy no. 1



Alloy no. 2



Alloy no. 3



Alloy no. 4

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Alloy no. 5



Alloy no. 6

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Alloy no. 7



Alloy no. 8

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Alloy no. 9



Alloy no. 10

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Alloy no. 11



Alloy no. 12
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Lysis index (cont.)



Alloy no. 13



Alloy no. 14

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Lysis index (cont.)



Alloy no. 15



Alloy no. 16

APPENDIX F

EFFECF OF Al AND Ni CHART

Thermal properties



Mechanical properties



Mechanical properties (cont.)



Static immersion: Elution of elements





Cyclic immersion: Colour, lightness, chromaticness difference

Potentiodynamics



APPENDIX G STATISTICS

1. Mechanical properties

Multivariate Tests

Effect		Value	F	Hypothesis df	Error df	Sig.
Intercept	Pillai's Trace	1.0	60606.5	5	48	0.000
	Wilks' Lambda	0.0	60606.5	5	48	0.000
	Hotelling's Trace	6313.2	60606.5	5	48	0.000
	Roy's Largest Root	6313.2	60606.5	5	48	0.000
Aluminum	Pillai's Trace	1.7	12.6	15	150	0.000
	Wilks' Lambda	0.0	54.0	15	132.9	0.000
	Hotelling's Trace	88.4	275.1	15	140	0.000
	Roy's Largest Root	87.1	870.9	5	50	0.000
Nickel	Pillai's Trace	2.0	20.0	15	150	0.000
	Wilks' Lambda	0.0	288.9	15	132.9	0.000
	Hotelling's Trace	1704.7	5303.6	15	140	0.000
	Roy's Largest Root	1697.1	16971.1	5	50	0.000
Al * Ni	Pillai's Trace	2.2	6.6	30	260	0.000
	Wilks' Lambda	0.0	11.0	30	194	0.000
	Hotelling's Trace	10.9	16.9	30	232	0.000
	Roy's Largest Root	8.4	72.8	6	52	0.000

Tests of Between-Subjects Effects

Source	Dependent Variable	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected	Elongation	10830.2	12	902.5	32.3	0.000
Model	Tensile strength	1313203.9	12	109433.7	78.1	0.000
	Surface hardness	287250.5	12	23937.5	8235.3	0.000
	Modulus of elasticity	33644.3	12	2803.7	20.8	0.000
	Proof strength	929431.9	12	77452.7	839.3	0.000
Intercept	Elongation	43312.4	1	43312.4	1548.5	0.000
	Tensile strength	7731335.8	1	7731335.8	5516.8	0.000
	Surface hardness	869349.0	1	869349.0	299085.0	0.000
	Modulus of elasticity	546932.5	1	546932.5	4048.7	0.000
	Proof strength	1898494.3	1	1898494.3	20573.2	0.000
Aluminum	Elongation	1222.3	3	407.4	14.6	0.000
	Tensile strength	106531.2	3	35510.4	25.3	0.000
	Surface hardness	11154.5	3	3718.2	1279.2	0.000
	Modulus of elasticity	12743.1	3	4247.7	31.4	0.000

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Mechanical properties (cont.)

	Proof strength	72196.3	3	24065.5	260.8	0.000
Nickel	Elongation	9459.5	3	3153.2	112.7	0.000
	Tensile strength	1115711.9	3	371904.0	265.4	0.000
	Surface hardness	238452.9	3	79484.3	27345.2	0.000
	Modulus of elasticity	12510.1	3	4170.0	30.9	0.000
	Proof strength	741687.2	3	247229.1	2679.1	0.000

Tests of Between-Subjects Effects (cont.)

		Type III				
Source	Dependent Variable	Sum of Squares	df	Mean Square	F	Sig.
Al * Ni	Elongation	638.4	6	106.4	3.8	0.003
	Tensile strength	10533.6	6	1755.6	1.3	0.295
	Surface hardness	1136.8	6	189.5	65.2	0.000
	Modulus of elasticity	9582.9	6	1597.2	11.8	0.000
	Proof strength	3033.4	6	505.6	5.5	0.000
Error	Elongation	1454.5	52	28.0		
	Tensile strength	72873.1	52	1401.4		
	Surface hardness	151.1	52	2.9		
	Modulus of elasticity	7024.6	52	135.1		
	Proof strength	4798.6	52	92.3		
Total	Elongation	74215.8	65			
	Tensile strength	9240105.9	65			
	Surface hardness	1006841.3	65			
	Modulus of elasticity	724098.0	65			
	Proof strength	2394358.4	65			
Corrected		12284.7	64			
Total	Tensile strength	1386077.0	64			
	Surface hardness	287401.7	64			
	Modulus of elasticity	40668.9	64			
	Proof strength	934230.5	64			

Multivariate Tests						
Effect		Value	F	Hypothesis df	Error df	Sig.
Intercept	Pillai's Trace	1.0	3627205.1	4	141	0.000
	Wilks' Lambda	0.0	3627205.1	4	141	0.000
	Hotelling's Trace	102899.4	3627205.1	4	141	0.000
	Roy's Largest Root	102899.4	3627205.1	4	141	0.000
Aluminum	Pillai's Trace	2.8	438.1	12	429	0.000
	Wilks' Lambda	0.0	4972.7	12	373.3	0.000
	Hotelling's Trace	1006.0	11709.0		419	0.000
	Roy's Largest Root	819.2	29287.7	4	143	0.000
Nickel	Pillai's Trace	2.3	115.3	12	429	0.000
	Wilks' Lambda	0.0	6842.7	12	373.3	0.000
	Hotelling's Trace	20168.9	234744.1	12	419	0.000
	Roy's Largest Root	20114.7	719101.0	4	143	0.000
Al * Ni	Pillai's Trace	3.5	104.8	36	576	0.000
	Wilks' Lambda	0.0	1100.4	36	530.1	0.000
	Hotelling's Trace	1732.9	6715.0	36	558	0.000
	Roy's Largest Root	1582.6	25322.0	9	144	0.000

2. Static immersion: Colour, lightness and chromaticness difference

Tests of Between-Subjects Effects

Source	Dependent Variable	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected	DE	28595.0	15	1906.3	72507.0	0.000
Model	DL	23870.6	15	1591.4	197379.6	0.000
	Da	906.9	15	60.5	643.0	0.000
	Db	7567.3	15	504.5	9400.3	0.000
Intercept	DE	135594.4	1	135594.4	5157314.0	0.000
	DL	108040.4	1	108040.4	13400363.8	0.000
	Da	4419.5	1	4419.5	46998.1	0.000
	Db	19479.0	1	19479.0	362962.4	0.000
Aluminum	DE	774.2	3	258.1	9815.5	0.000
	DL	743.8	3	247.9	30752.5	0.000
	Da	387.5	3	129.2	1373.7	0.000
	Db	1119.5	3	373.2	6953.3	0.000
Nickel	DE	25090.8	3	8363.6	318108.7	0.000
	DL	21385.6	3	7128.5	884160.5	0.000
	Da	321.4	3	107.1	1139.2	0.000
	Db	4452.7	3	1484.2	27656.6	0.000
Al * Ni	DE	2729.9	9	303.3	11537.0	0.000
	DL	1741.1	9	193.5	23995.0	0.000
	Da	198.0	9	22.0	234.0	0.000
	Db	1995.1	9	221.7	4130.6	0.000
Error	DE	3.8	144	0.0		
	DL	1.2	144	0.0		
	Da	13.5	144	0.1		
	Db	7.7	144	0.1		
Total	DE	164193.1	160			
	DL	131912.2	160			
	Da	5339.9	160			
	Db	27054.0	160			

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BIOGRAPHY

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