INFLUENCE OF REDUCTION RATIO OF CROSS SECTIONAL AREA IN DRAWING STAINLESS STEEL WIRE FOR ORTHODONTIC USE

SIRIWAT CHAMNUNPHOL

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE (ORTHODONTICS) FACULTY OF GRADUATE STUDIES MAHIDOL UNIVERSITY 2008

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	Mr. Siriwat Chamnunphol Candidate
	Assoc. Prof. Niwat Anuwongnukroh, D.D.S., M.S.D. (Orthodontics), Diplomate Thai Board of Orthodontics Diplomate American Board of Orthodontics Major-Advisor
	Assoc.Prof. Surachai Dechkunakorn, B.Sc., D.D.S., Dip.in Orthodontics Diplomate Thai Board of Orthodontics Co-Advisor
	Assoc.Prof. Pongpan Kaewtatip, D. Eng Co-Advisor
Prof. Banchong Mahaisavariya, M.D. Dean Faculty of Graduate Studies	Assoc.Prof. Nita Viwattanatipa, D.D.S., M.S.(Orthodontics), Diplomate American Board of Orthodontics Chair Master of Science Programme in Orthodontics Faculty of Dentistry

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was submitted to the Faculty of Graduate Studies, Mahidol University For the degree of Master of Science (Orthodontics)

> on March 24, 2008

	Mr. Siriwat Chamnunphol Candidate
	Dr.Julathep Kajornchaiyakul,, Ph.D. Chair
	Assoc. Prof. Niwat Anuwongnukroh, D.D.S., M.S.D. (Orthodontics), Diplomate Thai Board of Orthodontics Diplomate American Board of Orthodontics Member
Assoc.Prof. Surachai Dechkunakorn, B.Sc., D.D.S., Dip.in Orthodontics Diplomate Thai Board of Orthodontics Member	Assoc.Prof. Pongpan Kaewtatip, D.Eng., Member
Prof.Banchong Mahaisavariya, M.D. Dean Faculty of Graduate Studies Mahidol University	Assoc. Prof. Theeralaksna Suddhasthira, Ph.D. Dean Faculty of Dentistry Mahidol University

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Siriwat Chanunphol

INFLUENCE OF REDUCTION RATIO OF CROSS SECTIONAL AREA IN DRAWING STAINLESS STEEL WIRE FOR ORTHODONTIC USE

SIRIWAT CHAMNUNPHOL 4937193 DTOD/M

M.Sc. (ORTHODONTICS)

THESIS ADVISORS: NIWAT ANUWONGNUKROH, D.D.S., M.S.D. (ORTHODONTICS), DIPLOMATE THAI BOARD OF ORTHODONTICS, SURACHAI DECHKUNAKORN B.SC, D.D.S., DIPLOMATE THAI BOARD OF ORTHODONTICS, PONGPAN KAEWTATIP D.ENG

ABSTRACT

Stainless steel wires, especially Austenitic stainless steel, are widely used in orthodontics. In Thailand, commercial orthodontic stainless steel wires are imported from oversea and this makes them expensive. Locally general purpose stainless steel wires usually have larger diameter and are not suitable for orthodontic use. It is interesting to study the general purpose stainless steel wire by reduction its size and to test whether it is comparable to commercial orthodontic stainless steel wire.

This research studied 0.5 mm. round commercial orthodontic stainless steel wires and general purpose stainless steel wires sold in the market in order to compare physical, mechanical and chemical properties of each type of wire and to construct a reference for manufacturing of wires that can be used in orthodontics. The experiment also studied the effects of cold work wire drawing on mechanical and chemical properties of wires. Three sizes of stainless steel wire type 304 were drawn. Wires with diameters of 0.55, 0.6, and 0.725 mm. were reduced to 0.5 mm. using reduction ratios of 20%, 30% and 50%, respectively. Single drawing process was used in drawing with 20% and 30% reduction ratios, while double drawing process was used in drawing with 50% reduction ratio. Drawing speed was 0.41 mm/s using a drawing die made from tungsten carbide with an approach angle of the die at 12 degrees.

Experimental results indicated no statistically significant difference (p>0.05) in mechanical properties in terms of stiffness and modulus of elasticity among orthodontic, general purpose and drawn wires. However, in terms of yield strength, ultimate tensile strength and % of elongation, the statistically significant differences (p<0.05) were found across the 3 types of wires. Increasing in % reduction ratio of cross sectional area in wire drawing leads to more strength and less % elongation. As for physical properties, the wire with smoothest surface was the orthodontic wire, followed by the general purpose stainless steel wires, and the lowest smoothness was found in the drawn wires. Furthermore, study of chemical properties revealed that corrosion and rust were found in drawn wires with all 3 reduction ratios.

KEY WORDS: REDUCTION RATIO / STAINLESS STEEL / WIRE DRAWING / ARCHWIRE

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อิทธิพลของอัตราการลดขนาดหน้าตัดในการดึงลวดเหล็กกล้าไร้สนิมสำหรับทันตกรรมจัด ฟัน(INFLUENCE OF REDUCTION RATIO OF CROSS SECTIONAL AREA IN DRAWING STAINLESS STEEL WIRE FOR ORTHODONTIC USE)

ศิริวัฒน์ ชำนาญผล 4937193 DTOD/M

วท.ม. (ทันตกรรมจัดฟัน)

กณะกรรมการควบคุมวิทยานิพนธ์ : นิวัต อนุวงศ์นุเกราะห์, D.D.S., M.S.D. (ORTHODONTICS), Diplomate Thai Board of Orthodontics, สุรชัย เดชคุณากร, B.Sc, D.D.S., Diplomate Thai Board of Orthodontics, พงศ์พันธ์ แก้วตาทิพย์, D.Eng.

บทคัดย่อ

้ถวดสแตนเลส สติลได้ถูกนำมาใช้อย่างกว้างขวางในทางทันตกรรมจัดฟัน โดยเฉพาะ ้ชนิด ออสเทนนิติก ซึ่งต้องนำเข้าจากต่างประเทศและมีรากาสูง อีกทั้งที่มีจำหน่ายในประเทศ ้จะมีขนาคเส้นผ่าศูนย์กลางที่ใหญ่ไม่เหมาะกับการนำมาใช้ในผู้ป่วย จุดประสงค์วิจัยนี้เพื่อ ศึกษาลวดสแตนเลส สติล ทางทันตกรรมจัดฟันชนิดกลมขนาด 0.5 มม. และลวดสแตนเลส ิสตีล ชนิดทั่วไปขนาดเดียวกันที่มีขายตามท้องตลาด โดยการเปรียบเทียบ คุณสมบัติทาง ้กายภาพ ทางกลและทางเคมี เพื่อนำผลที่ได้มาเป็นมาตรฐานในการผลิตเส้นลวดที่สามารถใช้ ในทางทันตกรรมจัดฟันได้ พร้อมทั้งศึกษาอิทธิพลของการรีดเย็นต่อคุณสมบัติทางกายภาพ ทางกล และทางเคมี วิธีการทำโดยดึงลวดสแตนเลส สตีล ชนิด 304 สามขนาด ที่มี เส้นผ่าศนย์กลาง 0.55 0.6 และ 0.725 มม.ให้มีขนาคลคลงเหลือ 0.5 มม. มีอัตราการลด ้ขนาดหน้าตัดของลวด 20%, 30% และ 50% ตามลำดับใช้กระบวนการดึงลวดแบบขั้นเดียว ในการถดขนาดหน้าตัดของถวด 20% และ 30% ส่วนการถดขนาดหน้าตัดถวด 50% จะใช้ กระบวนการคึงลวดแบบสองขั้นใช้ความเร็วในการคึง 0.41 มม./วินาที แม่พิมพ์ดึงลวดทำจาก ทั้งสเตนการ์ไบด์ ซึ่งมีมุมไหลเข้าเท่ากับ 12 องศา ผลการศึกษาพบว่า ลวดทันตกรรมจัดฟัน ้ถวดสแตนเถส สตีลทั่วไปและถวดที่ดึงลดขนาดทั้งสาม ไม่มีความแตกต่างของคุณสมบัติทาง กลอย่างมีนัยสำคัญทางสถิติ(p<0.05)ในส่วนของก่ากวามแข็ง มอดูลัสยึดหยุ่น ส่วนก่ากวาม แข็งแรงกราก กวามแข็งแรงสูงสุดและร้อยละของกวามยืดของวัสดุ พบว่ามีกวามแตกต่างกัน ้อย่างมีนัยสำคัญทางสถิติ(p<0.05)ในทั้งสามกลุ่ม การถดขนาดหน้าตัดของถวดด้วยอัตราที่ ้มากขึ้นทำให้ถวดมีความแข็งแรงมากขึ้นในขณะที่ร้อยละของความยืดลดลง ส่วนคณสมบัติ ทางกายภาพพบว่าถวดทันตกรรมจัดฟันจะมีผิวที่เรียบมากที่สุด รองลงมาเป็นลวดสแตนเลส ้ทั่วๆไปและลวคที่ดึงลดขนาดตามลำดับ และในด้านคุณสมบัติทางเคมีพบว่าลวคในกลุ่มที่ดึง ้ถดขนาดเองจะเกิดการกัดกร่อนเป็นสนิมเหล็กสังเกตได้ชัดเจนในทั้งสามอัตราการถดขนาด หบ้าตัด

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CHAPTER I INTRODUCTION

Stainless steels (S.S) are most frequently used materials for orthodontic treatment with fixed appliances. Over several decades, engineering technologies have developed over 100 different compositions of SS, used in many applications and various fields. The alloy of SS most frequently used in contemporary medical practice is American Iron and Steel Institute (AISI) type 316L, which contains molybdenum and thus is more resistant to pit corrosion and AISI type 304 used for orthodontic materials. It is not clearly known what the composition is according to the manufacturers' information leaflets of orthodontic SS materials and, even more important, how the alloys were processed. This is important because the specific handling of the SS during production significantly determines its mechanical and physical properties.

The term stainless steel is applied to all alloys of iron and carbon which contain chromium, nickel, or other elements which impart to the steel for the property of resisting corrosion. There are over forty stainless steel alloys whose properties vary greatly. The three main groups are martensitic, ferritic, and austenitic. The steels used in orthodontics come from the austenitic group. The most widely used 18:8 alloy contains approximately 18 percent chromium, 8 percent nickel, 0.2 percent carbon, and a trace of stabilizing elements. All of the austenitic steels have good corrosion resistance, hardness, yield and tensile strength. However, small sizes of wire usually demonstrate these qualities with quite a large degree of variation.

Austenitic steels are nonmagnetic unless heavily cold-worked, which results in slight magnetism. Metallurgists do not agree on the reason the stainless steels resist corrosion. It is generally believed that this resistance is due to the presence of a hydrous oxide film which is stabilized by chromium. This film forms naturally on the surface of the wire upon exposure to a suitably oxidizing environment. The film, which varies in composition from alloy to alloy, cannot be seen microscopically, is transparent and insoluble. If its continuity is broken by welding, soldering, or

mechanical working, it will reform naturally within a short period of time. Its formation may be hastened by exposure to a strong oxidizing agent. If the metal is to be exposed immediately to saliva, the continuity of the film can be restored by a process called pasivation.

Nowadays in Thailand, commercial orthodontic stainless steel wires are imported from oversea. It is interesting to study mechanical, physical and chemical properties of various imported orthodontic wires in Thai market. Additionally, it would be beneficial if orthodontic wire could be locally manufactured in Thailand. Therefore, this project will also study the influence of reduction ratio of cross sectional area in drawing of general purpose stainless steel wire (AISI 304) and compare their properties with those of commercial orthodontic wires.

CHAPTER II OBJECTIVES

This study is designed to investigate and compare the in vitro mechanical, physical and chemical properties of commercial orthodontic, general purpose and modified stainless steel wires by measuring their diameter, composition, yield strength, ultimate tensile strength , stiffness, % elongation, modulus of elasticity, surface hardness, surface roughness and corrosion.

The purposes are :

1. To investigate mechanical, physical and chemical properties of commercial orthodontic wire and general purpose wires.

2. To investigate influences of reduction ratio of cross sectional area on mechanical, physical and chemical properties.

3. To compare modified drawn wire with commercial orthodontic wire and general purpose wire.

Thesis questions :

1. What are the mechanical, physical and chemical properties of commercial orthodontic wire and commercial general purpose wires?

2. Does the % reduction ratio of cross sectional area affect the mechanical, physical and chemical properties of wires?

3. Do properties of the modified drawn wire comparable to commercial orthodontic wire and commercial general purpose wires?

Limitations of the study are:

1. This is an in vitro study, therefore, the result may not correspond to those found under intraoral conditions. However, the results may give some information in choosing commercial stainless steel orthodontic arch wires for clinical use and for producing stainless steel orthodontic arch wire in the future.

2. The result of the study may not be comparable to other studies due to the differences in the study design and material used, etc.

Expected benefits from the study are:

1. To know mechanical, physical and chemical properties of commercial orthodontic and commercial general purpose wire.

2. To know influences of reduction ratio of cross sectional area in wire drawing process on mechanical, physical and chemical properties.

3. If the results obtained from this study are positive, it may encourage the local production of orthodontic wire. This will not only help the economy grow by reduction of imported orthodontic wires but also provide fundamental knowledge of wire drawing in another diameter of orthodontic wires.

CHAPTER III LITERATURE REVIEW

The properties of materials determine their usefulness. In the context in which it is frequently used, the term "property" connotes something that a material inherently possesses. More properly, a property should be regarded as the response of a material to a given set of imposed conditions (e.g., temperature and/or pressure). Material properties are the link between the basic structure and composition of the material and the service performance of a part or component.

A wide variety of properties must be considered when choosing a material and a combination of properties is usually required for a given application. The properties of greatest importance for metals include [1] :

Physical properties, such as mass characteristics, thermal, electrical, magnetic, and optical properties.

Chemical properties, such as corrosion and oxidation resistance.

Mechanical properties, such as tensile and yield strength, elongation (ductility), toughness, and hardness.

Introduction of stainless steel

Stainless Steels

Iron-chromium steels, with possible additions of nickel and molybdenum, in combination with low carbon contents, are designated as "stainless steels" when a minimum of 12 % Cr is present to provide a passive layer of chromium oxide on the surface. This passive layer is responsible for the high corrosion resistance realized in stainless steels. Stainless steel was traditionally made in small electric arc furnace (EAF) by melting steel scrap, nickel and ferrochrome before the advent of oxygen refining. The modern practice of making stainless steel is based on a two-stage process. The first stage employs a conventional EAF for the rapid melting of scrap and ferroalloys but uses cheap high-carbon ferrochrome as the main source of

chromium. Because stainless steel manufacturing involves more scrap melting and alloying and less refining, EAFs are preferred over the oxygen based converter processes due to high external energy loads. The high-carbon melt prepared in an EAF is then refined in a second stage, using either argon- oxygen degassing (AOD) or by blowing with oxygen under vacuum oxygen decarburization (VOD). The AOD process currently produces over 80% of the stainless steel tonnage worldwide. Special desulfurizing slags are used in AOD where intimate metal-slag mixing can be achieved using argon stirring. Oxygen is capable of decarburizing the melt to less than 0.01% C, and hydrogen levels are below 2 to 3 ppm. Sensitization in austenitic stainless steels leading to intergranular corrosion is markedly influenced by the presence of elongated particles or clusters of second phases, such as sulfides of other inclusions. The presence of nitrogen in some niobium-bearing stainless grades leads to carbonitride formation, which also deleteriously influences sensitization. Control of gaseous inclusions as well as sulfur are important in refining of stainless steels.

Stainless steels require expensive alloying additions of chromium, nickel, and molybdenum. Therefore, recovery of these elements needs special attention. Efficient slag reduction with stoichiometric amounts of silicon or aluminum permits overall recoveries of 97 to 100% for most metallic elements. Chromium recovery averages approximately 97.5%, and nickel and molybdenum recoveries are approximately 100%. Casting is usually done in a continuous caster for better productivity, although ingot casting and primary rolling is still more common for stainless steels than carbon steels. The cost of ferrochrome production affects stainless steel prices directly.

Commercial varieties of stainless steels are classified as austenitic (work hardenable), ferritic (work hardenable), austenitic-ferritic(duplex), or martensitic (hardenable by heat treatment). Although this classification is based on microstructure, it relates to two primary roles of alloy additions: (1) the balance between austenite formers (N, C, Ni, Co, Cu, and Mn) and ferrite formers (W, Si, Mo, Cr, V, and Al) controlling the high-temperature microstructure and (2) the overall alloy content, which controls the martensite transformation range, M_s - M_f , and the degree of martensite transformation at ambient temperature.

Stainless steels have lower thermal conductivity than carbon or alloy steels below 815 °C (1500 °F) and, therefore, need special attention in heating below 815 °C

(1500 °F) to avoid surface burning. In addition, hot-working temperature ranges for stainless steels are narrower than for carbon steels, requiring better temperature control during soaking and rolling. Martensitic grades are slow cooled or annealed after rolling because they are air hardening. Ferritic grades are finish rolled to lower temperatures to prevent grain growth that could lead to tearing and cracking. Austenitic grades require more rolling-mill power because they are stronger than ferritic grades and are also susceptible to grain growth. Sulfur control in reheating furnace atmospheres is important for austenitic grades due to the presence of nickel. Liquid nickel sulfide formation at the grain boundaries during rolling can lead to tears and cracks. Cold rolling of stainless steel has two primary objectives is reduction of hot rolled gage and cold forming into components. Except the high-carbon grades, all stainless steels are an enable to cold working. Pickling is performed following hot rolling.

The properties of carbon and alloy steels are dependent on the relationships between chemical composition processing, and microstructure. In this article, emphasis is placed on the effect of composition (alloying).

Alloying elements are added to ordinary (plain carbon) steels to modify their behavior during thermal processing (heat treatment of thermomechanical processing), which in turn results in improvement of the mechanical and physical properties of the steel. Specifically, alloying additions are made for one or more of the following reasons:

- 1. Improve tensile strength without appreciably lowering ductility
- 2. Improve toughness
- 3. Increase hardenability which permits the hardening of larger sections then possible with plan carbon steels or allows successful quenching with less drastic cooling rates, reducing the hazard of distortion and quench cracking
- 4. Retain strength at elevated temperatures
- 5. Obtain better corrosion reststance
- 6. Improve wear resistance
- 7. Impart a fine grain size to the steel

A semantic distinction can be made between alloying elements and residual elements ; the latter are not intentionally added to the steel, but result from the raw

materials and steelmaking practices used to produce the steel. Any particular element can be either alloying or residual. For example, some nickel or chromium could come into steel through alloy steel scrap and so be considered residual ; however, if either of these elements must be added to a steel to meet the desired composition range it might be considered an alloying element.

Both alloying and residual elements can profoundly affect steel production, manufacture into end products, and service performance of the end product. The effects of one alloying element on a steel may be affected by the presence of other elements ; such interactive effects are complex. In addition, the effects of a particular element may be beneficial to steel in one respect but detrimental in others.

Effects of alloying elements

General effects of various alloying elements commonly found in carbon and low-alloy steels are summarized below.

Carbon is the most important single alloying element in steel. It is essential to the formation of cementite (and other carbides). Pearlite, spheroidite (an aggregate of spherical carbides in a ferrite matrix), bainite and iron-carbon martensite. Microstructures comprising one or more of these components can be fabrication characteristics. The relative amounts and distributions of these elements can be manipulated by heat treatment to alter the microstructure, and therefore the properties of a particular piece of steel. Much of ferrous metallurgy is devoted to the various structures and transformations in ironcarbon alloys ; many other alloying elements are considered largely on the basis of their effects on the iron-carbon system.

Assuming that the comparisons are made among steels having comparable microstructures, the strength and hardness are raised as the carbon content is increased ; however, toughness and ductility are reduced by increases in carbon content (workability, weldability, and machinability are also deleteriously affected by higher carbon contents). The hardness of iron-carbon martensite is increased by raising the carbon content of steel, reaching a maximum at about 0.6% C. Increasing the carbon content also increases hardenability.

The amount of carbon required in the finished steel limits the type of steel that can be made. As the carbon content of rimmed steel increases, surface quality becomes impaired. Killed steels in approximately the 0.15 to 0.30% C content level may have poorer surface quality and require special processing to obtain surface quality comparable to steels with higher or lower carbon content. Carbon has a moderate tendency to segregate, and carbon segregation is often more significant than the segregation of other elements.

Manganese is normally present in all commercial steels. It is important in the manufacture of steel because it deoxidizes the melt and facilitates hot working of the steel by reducing the susceptibility to hot shortness. Manganese also combines with sulfur to form manganese sulfide stringers, which improve the machinability of steel. It contributes to strength and hardness, but to a lesser degree than does carbon ; the amount of increase depends on the carbon content. Manganese has a strong effect of increasing the hardenability of a steel.

Manganese has less of a tendency toward macrosegregation than any of the common elements. Steels with more than 0.60% Mn cannot be readily rimmed. Manganese is beneficial to surface quality in all carbon ranges (with the exception of extremely low-carbon rimmed steels).

Silicon is one of the principal deoxidizers used in steelmaking. The amount of this element in a steel, which is not always noted in the chemical composition specifications, depends on the deoxidation practice specified for the product. Rimmed and capped steels contain minimal silicon, usually less than 0.05%. Fully killed steels usually contain 0.15 to 0.30% silicon for deoxidation ; if other deoxidants are used, the amount of silicon in the steel may be reduced. Silicon has only a slight tendency to segregate. In low carbon steels, silicon is usually detrimental to surface quality, and this condition is more pronounced in low-carbon resulfurized grades.

Silicon slightly increases the strength of ferrite, without causing a serious loss of ductility. In larger amounts, it increases the resistance of steel to scaling in air (up to about 260 °C, or 500 °F) and decreases the magnetic hysteresis loss. Such high-silicon steels are generally difficult to process.

Copper has moderate tendency to segregate, and in appreciable amounts, it is detrimental to hot-working operations. Copper adversely affects forge welding, but it does not seriously affect arc or oxyacetylene welding. Detrimental to surface quality, copper exaggerates the surface defects inherent in resulfurized steels. Copper is, however, beneficial to atmospheric corrosion resistance when present in amounts exceeding 0.02%. Steels containing these levels of copper are referred to as weathering steels.

Chromium is generally added to steel to increase resistance to corrosion and oxidation, to increase hardenability, to improve high-temperature strength, or to improve abrasion resistance in high-carbon compositions. Chromium is a strong carbide former. Complex chromium-iron carbides go into solution in austenite slowly; therefore, a sufficient heating time before quenching is necessary.

Chromium can be used as a hardening element and is frequently used with a toughening element such as nickel to produce superior mechanical properties. At higher temperatures, chromium contributes increased strength ; it is ordinarily used for applications of this nature in conjunction with molybdenum.

Nickel, when used as an alloying element in constructional steels, is a ferrite strengthener. Because nickel does not form any carbide compounds in steel, it remains in solution in the ferrite, thus strengthening and toughening the ferrite phase. Nickel steels are easily heat treated because nickel lowers the critical cooling rate. In combination with chromium, nickel produces alloy steels with greater hardenability, higher impact strength, and greater fatigue resistance than can be achieved in carbon steels. Nickel alloy steels also have superior low-temperature strength and toughness.

Austenitic stainless steels

Characteristics and Compositions

Austenitic stainless steels constitute the largest stainless family in terms of number of alloys and usage. Like the ferritic alloys, they cannot be hardened by heat treatment. However, their similarity ends there. The austenitic stainless steels are essentially nonmagnetic in the annealed condition and can be hardened only by cold working. They usually possess excellent cryogenic properties and good high-temperature strength and oxidation resistance. Chromium content generally varies from 16 to 26% ; nickel content is less than or equal to approximately 35% ; and manganese content is less than or equal to 15%. The 200 series steels contain nitrogen, 4 to 15% Mn, and lower nickel contents (up to 7% Ni). The 300 series steels contain larger amounts of nickel and up to 2% Mn. Molybdenum, copper, silicon, aluminum,

titanium, and niobium can be added to confer certain characteristics, such as halide pitting resistance or oxidation resistance.

Properties and Applications.

The yield strengths of chromium-nickel austenitic stainless steels are rather modest and are comparable to those of mild steels. Typical minimum mechanical properties of annealed 300 series steels are yield strengths of 205 to 275 MPa (30 to 40 ksi), ultimate tensile strengths of 520 to 760 Mpa (75 to 110 ksi), and elongations of 40 to 60%. Annealed 200 series alloys have higher yield strengths ranging from 345 to 480 MPa (50 to 70 ksi). Higher strengths are possible in cold-worked forms, especially in drawn wire, in which a tensile strength of 1200 MPa (175 ksi) or higher is possible. The 200 series have work-hardening characteristics similar to types 301 and 302. Even the leanest austenitic stainless steels (e.g., types 302 and 304) offer general corrosion resistance in the atmosphere, in many aqueous media in the presence of foods, and in oxidizing acids such as nitric acid. Types 321 and 347 are essentially type 304 with additions of either titanium or niobium, respectively, which stabilize carbides against sensitization. The addition of molybdenum in types 316/316L provides pitting resistance in phosphoric and acetic acids and dilute chloride solutions, as well as corrosion resistance in sulfurous acid. An even higher molybdenum content, as in type 316L (3%), and even richer alloys further enhance pitting resistance. Nitrogen is added to enhance strength at room temperature and, especially, at cryogenic temperatures (e.g., type 304N). Nitrogen is also added to reduce the rate of chromium carbide precipitation and, therefore, the susceptibility to sensitization. It is also added to molybdenum-containing alloys to increase resistance to chloride-induced pitting and crevice corrosion. Higher amounts of chromium and/or nickel are used to enhance high-temperature oxidation resistance (e.g., types 309, 310 and 330). Copper and nickel can be added to improve resistance to reducing acids, such as sulfuric acid (type 320). Some of the more corrosion-resistant alloys, such as N08020 (20Cb-3) have nickel contents high enough (32 to 37%) to rate UNS classification as nickelbase alloys. Alloys containing nickel, molybdenum (~6%), and nitrogen (0.15 to 0.25%) are sometimes referred to as "superaustenitics,". These alloys were developed for improved resistance to chloride corrosion.

Heat treating of stainless steels

Heat treating of stainless steel produces changes in physical condition. Mechanical properties, and residual stress level and restores maximum corrosion resistance when that property has been adversely affected by previous fabrication or heating. Frequently, satisfactory corrosion resistance and optimum mechanical properties are obtained in the same heat treatment.

Austenitic Stainless Steels

Austenitic stainless steels can be divided into five groups:

- Conventional austenitics, such as types 301, 302, 303, 304, 305, 308, 309, 310, 316, and 317
- 2. Stabilized compositions. Primarily types 321, 347, and 348
- 3. Low-carbon grades, such as types 304L, 316L, and 317L
- 4. High-nitrogen grades, such as AISI types 201, 202, 304N, 316N, and the Nitronic series of alloys
- Highly alloyed austenitics, such as 317LM, 317LX, JS700, JS777, 904L, AL-4X, 2RK65, Carpenter 20Cb-3. Sanicro 28, AL-6X, AL-6XN, and 254 SMO

Annealing

Conventional austenitics cannot be hardened by heat treatment but will harden as a result of cold working. These steels are usually purchased in an annealed or coldworked state. Following welding or thermal processing, a subsequent anneal may be required for optimum corrosion resistance, softness, and ductility. During annealing, chromium carbides, which markedly decrease resistance to intergranular corrosion, are dissolved. Annealing temperatures, which vary some what with the composition of the steel.

Because carbide precipitation can occur at temperatures between 425 and 900°C (800 and 1600 °F). The annealing temperature should be safely above this limit. Moreover, because all carbides should be in solution before cooling begins, and because the chromium carbide dissolves slowly, the highest practical temperature consistent with limited grain growth should be selected. This temperature is in the vicinity of 1095 °C (200 °F).

Cooling from the annealing temperature must be rapid, but it must also be consistent with distortion limitations. Whenever distortion considerations permit, water quenching is used, thus ensuring that dissolved carbides remain in solution. Because type 310 precipitates carbides more rapidly, this material invariably requires water quenching. Where distortion considerations rule out such a fast cooling rate, cooling in an air blast is used. With some thin-section parts, even this intermediate rate of cooling produces excessive distortion, and parts must be cooled in still air. If still air does not provide a cooling rate sufficient to prevent carbide precipitation, maximum corrosion resistance will not be obtained. A solution to this dilemma is the use of a stabilized grade of one of the low-carbon alloys.

Orthodontic stainless steel wire

The properties of an ideal wire material for orthodontic purposes [2] can be described largely in terms of these criteria: it should posses (1) high strength, (2) low stiffness (in most application), (3) high range, and (4) high formability. In addition, the material should be weldable or solderable, so that hooks or stops can be attached to the wire. It should also be reasonable in cost. In contemporary practice, no one arch wire material meets all these requirements, and the best results are obtained by using specific arch wire materials for specific purposes.

After the World War I, stainless steel became widely available, with its manufacturers pushing for new markets and offering technical assistance. Angle used it in his last year(1930) as ligature wire. By 1937 the value of stainless steel as an orthodontic material has been confirmed [3].

The stainless steel alloys used for orthodontic wire are of the "18-8" austenitic type, containing approximately 18 % chromium and 8 % nickel. While a report several decades ago showed that a 17-7 precipitation –hardenable stainless steel alloy had higher yield strength and greater resilience in bending than the commonly used stainless wire alloys, this alloy never achieved commercial popularity for orthodontic wires[4].

The chromium in the stainless steel forms a thin, adherent passivating oxide layer that provides corrosion resistance by blocking the diffusion of oxygen to the underlying bulk alloy. Approximately 12-13 wt% chromium is required to impart the

necessary corrosion resistance to these alloys. The chromium, carbon, and nickel atoms (and atoms of other metals in the composition) are incorporated into the solid solution formed by the iron atoms. Since the nickel atoms are not strongly bonded to form some intermetallic compound the likelihood of in vivo show nickel ion release from the alloy surface is increased, which may have implications for biocompatibility of these alloys.

Wire drawing process[5]

Drawing operations involve the forceing of metal through a die by means of a tensile force applied to the exit side of the die. Most of the plastic flow is caused by compression force which arises from the reaction of the metal with the die. Usually the metal has a circular symmetry, but this is not an absolute requirement. The reduction in diameter of a solid bar or rod by successive drawing is known as bar, rod, or wire drawing, depending on the diameter of the final product.

Bar, wire and tube drawing are usually carried out at room temperature. However, because large deformations are usually involved, there is considerable temperature rise during the drawing operation.

The principles involved in the drawing of bars, rod, and wire are basically the same, although the equipment that is use is different for the different-size products. Rod and tube, which cannot be coiled, are produced on drawbenches. The rod is pointed with a swager, inserted through the die, and clamped to the jaws of the drawhead. The drawhead is moved either by a chain drive or by a hydraulic mechanism. Drawbenches with 300,000 Ib pull and 100 ft of runout are available. Draw speeds vary from about 30 to 300ft/min.

The cross section through a typical conical drawing die is shown in Figure 1. The entrance angle of the die is made large enough to allow room for the lubricant that adheres to the die. The apporach angle is the section of the die where the actual reduction in diameter occurs. The bearing surface serves to guide the rod or wire as it exits from the die. An important characteristic of a drawing die is the half-die angle, denoted by α . At the present time most drawing dies are made from tungsten carbide because it provides long die life.

Wire drawing starts with hot-rolled wire rod. The rod is first cleaned by

pickling to remove any scale which would lead to surface defects and excessive die wear. For the production of steel wire the next step consists of coating the wire rod with lime or plating it with a thin layer of copper or tin. The lime serves as an absorber and carrier of the lubricant during dry drawing, and it also serves to neutralize any acid remaining from pickling. In dry drawing the lubricant is grease or soap power while in wet drawing the entire die is immersed in a lubricating fluid of fermented rye-meal liquor or alkaline soap solution.



Figure 1 : conical drawing die

The electroplated coating of copper or tin is used in the wet drawing of steel wire. No coating is generally used for drawing copper wire. After surface preparation of the wire rod, it is pointed, passed through the die, and fastened to the draw block. For coarse wire, with a final diameter greater than ¹/₄ in., a single draw block, called a bull block, is used. For fine wire (Fig 2), a large number of draw blocks are used, with the wire passing through a number of dies until it is reduced to its final size in one continuous operation. For fine wire, reductions per pass of 15 to 25 percent are used, while for coarse wires the reduction per pass may be 20 to 50 percent. Drawing speeds of modern wire-drawing equipment may be over 5000 ft/min.

Nonferrous wire and low-carbon steel wire are produced in a number of tempers ranging from dead soft to full hard. Depending on the metal and the reductions involved, intermediate anneals may be required. Steel wire with a carbon content greater than 0.25 percent is given a special patenting heat treatment. This consists in heating above the upper critical temperature and then cooling at a controlled rate or transforming in a lead bath at a temperature around 600° F to cause the formation of fine pearlite. Patenting produces the best combination of strength and ductility for the successful drawing of high-carbon music and spring wire.



Figure 2 : Bull block for fine wire

Internal defects in rod and wire include internal cracks due to seams or pipe in the hot-rolled strating material and a defect know as cupping. Cupping is the rupturing of the center of the wire when it is subjected to a tensile force. It can be recognized by a localized necking during drawing or by a cup-and-cone type of fracture when the wire is broken. Problems from cupping are more frequent with large die angles and high friction. Surface checking may result from improper surface lubrication. Longitudinal scratches are caused by a scored die, by improper lubrication, or by abrasive particles being drawn into the die with the wire. Slivers and seams result from cold shuts and blowholes in the hot-rolled strating material. Surface discoloration and ground-in oxide result from improper cleaning of the hotrolled bar and rod.

The force required to draw a wire throught a die is the summation of the force required to decrease the diameter uniformly (as in tensile elongation), the force required to produce nonuniform shear deformation of the surface layers at the entry to and exit from the die (redundant work), and the force required to overcome the friction between the wire and the die wall. The first and last factors can be included without

too much difficulty in an analysis of the wire-drawing process, but the nonhomogeneous deformation presents a problem which has not yet been adequately solved. The total force required for wire drawing can be considered to depend on the following factors: (1) the die angle: (2) the percent reduction: (3) the flow stress of the material: (4) the die friction, which is a function of the die material, the lubrication, and the drawing speed.

Studies of the deformation of grids scribed on the longitudinal axis of drawn bars have demonstrated the nonhomogeneous deformation produced by drawing[6]. In Figure 3 shows that for a given reduction in diameter the amount of shear deformation in the direction opposite to the draw pull increases with increasing halfdie angle. Only the elements on the axis of the bar undergo pure elongation. For large die angles the large shear deformation results in an increased londitudinal tensile stress at the center of the wire which can exceed the fracture stress and can thereby result in the cupping type failure. For a given die angle the shear deformation becomes less important with increasing percent reductions. For this reason, theoretical treatments of wire drawing which neglect the nonuniform deformation have greater validity at high reductions. Because of the increased surface shear deformation, the yield and tensile strengths of drawn wires are higher for larger die angles. This effect is greater for lower reduction.



Figure 3 : Nonhomogeneous deformation in various die angle

Although the nonuniform shear deformation is lower with smaller die angles, the wall friction is higher. Since the draw force depends in a complicated way on the die angle, reduction, the flow stress, and the friction, there will be an optimum die angle which results in the minimum draw force for a given reduction. This optimum die angle will depend on the reduction, the lubrication, and the material involved. All other factors being constant, the optimum die angle increases with amount of reduction. The drawing speed has little effect on the drawing force. However, there is greater temperature rise with high drawing speeds, and lubrication becomes more difficult.

Another factor to consider is the existence of a back pull in the direction opposite to the draw pull. A back pull of considerable magnitude can arise from frictional forces acting on the draw blocks of multiple drawing machines, or it may be purposely applied for the reasons given below. A back pull materially increases the drawing force. On the other hand, it reduces the wall pressure in the die and reduces the friction, and therefore die wear is appreciably decreased.

Two distinct types of residual-stress patterns are found in cold-drawn rod and wire, depending upon the amount of reduction. For reductions per pass of less than about 1 percent the longitudinal residual stresses are compressive at the surface and tensile at the axis, the radial stresses are tensile at the axis and drop off zero at the free surface, while the circumferential stresses follow the same trend as the londitudinal residual stresses. For larger reductions of commercial significance the residual pattern. In this case the longitudinal stresses are tensile at the surface and compressive at the axis of the rod, the radial stresses are compressive at the axis, and the circumferential stresses follow the same pattern as the longitudinal stresses. The first type of residual-stress pattern is characteristic of forming operations where the deformation is localized in the surface layers.

The effect of die angle and the amount of reduction per pass on the longitudinal residual stress in cold-drawn brass wire was investigated by Linicus and Sachs[7], Figure 4, shows that for a given reduction the longitudinal residual stress increases with the half-die angle. Maximum values of residual stress are obtained for reduction in the region of 15 to 35 percent.

Fac. of Grad. Studies, Mahidol Univ.



Figure 4 : Effect of die angle and reduction ratio on residual stress

A study by Kaewtatip et al [8] reveals that the faster the speed of wire drawing, the lower the quality of wire surface and the higher the tendency to get strain hardening inside the wire resulting in higher ultimate tensile strength from the reduction of % elongation of wire after drawing. Research also points out that too wide approach angle of die can lead to the decrease in quality of wire surface and increase in strain hardening inside the wire.

Norasat et al [9] report that tensile strength in the wire will reduce to the lowest level at approach angle is 6 degree, while at 4 degree, tensile strength will rise to highest level. The study provides some thoughts that tensile strength within the wire will decline when the pressure of lubricant is high. Since the higher the pressure of lubricant, the lower of friction on the surface. Furthermore, high pressure of lubricant will contribute to high quality of wire surface after drawing process.

Wantang et al [10] attempt to improve the efficiency of stainless steel wire drawing process focusing on lubrication. By reducing diameter of wire to 2.30 mm. (or the reduction ratio of the process is 9 percent), drawing speed determined to be 0.12 m/s. drawing die made from tungsten carbide, an approach angle of the dies is 12

degree, the results showed that TiC and TiCN film that coat inner surface of die, exhibits high wear resistant and improve surface roughness. TiCN film exhibited a bright surface. The higher viscosity of lubricant exhibited a higher wear resistant and accurate diameter of finished wire. The result had shown the wires surface roughness depended on lubricant viscosity. The lower viscosity of lubricant, the better is the wire surface roughness.

Manufacturing of orthodontic wire [4]

The starting point for manufacturing of orthodontic wires is the casting of an ingot of having the appropriate alloy composition. This ingot is then subjected to a series of mechanical reduction operations until the cross section is sufficiently small for wire drawing. The drawing for a round wire must be performed in series of steps until the final desired diameter is achieved, since the alloy rapidly work-hardens during each step of this process. Generally, more than one company is involved in the complex wire manufacturing sequence.

Wire with rectangular cross section are manufactured from round wires, using a Turk's head apparatus having two pairs of rollers positioned at right angles. Accordingly, orthodontic wires with rectangular or square cross sections have some inevitable rounding at the corners. This can make an important contribution to the archwire-bracket torque delivery, particularly when there is relatively tight engagement of the wires in the bracket slots.

Heat treatments are necessary during wire manufacturing to eliminate the extensive work hardening that occurs during the various stages of mechanical reduction. Information about these heat treatments is proprietary to the manufactures, as are details about the reduction per pass, the number of passes, the die and die lubrication materials. Special atmospheric conditions are needed for manufacturing the titanium-containing orthodontic wire because of the reactivity of these alloys with air. There is a tendency for the titanium –containing alloys to bind to the die or roller surfaces, resulting in increased surface roughness compared to other wire alloys. This can significantly affect the archwire-bracket friction, as will be discussed later.

By the 1950s stainless steel alloys were used for most orthodontic wires. Stainless steel wires remain popular because of their favorable combination of low cost and excellent formability, along with good mechanical properties. These wires can be soldered and welded for the fabrication of complex appliances, although it is necessary to use solder to provide reinforcement to weld joints.

Heat Treatment of orthodontic stainless steel wire [11]

Stainless steel, because of its constitution, can be strengthened only by coldworking or plastic deformation. Thus in forming an arch wire, a very high tensile strength may be produced but its flexibility usually decreases in varying degrees. This is importance since the elastic strength is one of the factors governing resilience of the wire. These properties allow the storage of forces and their delivery to the teeth at various rates. In order to increase the resiliency of the wire, various methods of heat treatment have been proposed.

Kemler [12] found that a low-temperature heat treatment caused an increase in the proportional limit and modulus of resilience of chrome alloy wires. He found that optimum heat treatment was five to fifteen minutes at 700-800°F.

Backofen and Gales [13] stated that ten minutes at 750-820°F produced optimum results.

Funk [14] produced the most marked increase in elastic properties at 850°F for a period of three minutes. However, he stated that good results can be obtained with in a temperature latitude of approximately 200 °F.

In each of these methods a marked increase as much as 40 percent was produced in the resiliency, with corresponding improvements in the tensile strength and proportional limit. Although wires of smaller diameter absorb heat faster than wires of larger diameter, there is no significant difference in the results to warrant a time-temperature ratio for the various size-wires. Although heat treatments increase the elastic strength and resiliency of a wire, they produce only slight changes in the elastic modulus.

On the other hand, Carsten [15] state that the stiffness of wire is dependent on the modulus of elasticity and was shown to be uneffected by the heat treatment. He suggested that for maximum effect it should be performed at 350-375°C for 20-25 minutes in dental furnace.

Metallurgical report from the Unitek Corporation [16], showed that heat-

treatment increase in the modulus of elasticity which is reported most often as 2 to 5 percent, but in one case as 15 percent.

In other words, the treatments change the degree to which the metal may be deformed, but the force for a given deformation remains almost identical. An archwire which has its elastic qualities increased by heat treatment will be more likely to assume its original shape after distortion. Since it will have more resistance to permanent set after deflection than a wire which has not been heat treated, maximum force will be applied during the expected range of tooth movement. Stabilization and anchorage will be enhanced because the archwire will maintain its form over a longer period of time. Opening and closing loops will produce a more continuous force over a larger range of tooth movement.

In conjunction with improving the elastic qualities of stainless steel wire, the heat treatment relieves the stress which are retained from cold-working. After work-hardening any spring temper chrome alloy, there is a tendency for the wire to return to its original position. These internal stresses are usually unequally distributed throughout the wire after bending. Proper heat treatment will relieve these stresses sufficiently to cause a reduction in the amount of breakage seen in clinical use. When a force is applied to the archwire or spring, the total amount of stress present is equal to the residual stresses plus those produced by the force. If there is a great deal of residual stress present in the wire, plastic deformation may occur when only a small force is applied. If these residual stresses are removed, a greater force can be applied and a lager range of action will be present.

Howe and Crimmins [17] studied stress relief tests and showed that generally much less than half of the residual stress is removed in the range of conventional stress relief heat treatment(700° to 900°F for 5-15 minutes).

The degree of stress relief increases with increasing temperature. However, care must be taken not to approach the lower limits of the annealing range of the steel. This range commences at approximately 1100°F. If the wires are subjected to temperatures above 1100°F for even short periods of time, some degree of softening will occur along with reduction in the proportional limit and tensile strength.

Wilkinson [18] explained that time and temperature affect tensile strength and hardness in directly proportional. If softening of a stainless steel archwire occurs, heat

treatment procedures now in use are incapable of restoring its physical properties. The only way in which an annealed austenitic steel wire maybe strengthened is through cold-working.

When the wire becomes annealed due to overheating, recrystallization of the metal takes place. The long fibrous grains which were produced by rolling and drawing in the fabrication of the wire are transformed into large equiaxed grains. These large grains, all of which have approximately the same dimensions in all directions, are responsible for the softened state of the wire. This process is governed mainly by two factors, time and temperature. A fast rate of recrystallization occurs at higher temperatures. Once this has taken place, continued heating causes grains growth. The grain size can be reduced by alternate plastic deformation and recrystallization, care being taken not to prolong the heating.

If annealing is desired to relieve brittleness by producing a softened, more ductile wire, it can be brought about quickly by placing the material in an oven at a temperature of 2000 °F or by heating in a flame or with electrodes until red hot. The wire can then be easily manipulated; work -hardening will restore its temper.

In general, a more effective working appliance will be produced by the proper heat treatment. This procedure should be carried out only after the completion of all the necessary bends. Repeated checking of the effectiveness of the selected heat treatment procedure may be necessary at intervals since there is not as yet as satisfactory a level of quality control in the fabrication of stainless steel wire as has been attained in the manufacture of wrought gold wires. The effectiveness of the heat treatment is determined to a large extent by the composition of the steel. In many cases the exact chemical composition of the wire is not given and the physical properties are not clearly defined by manufacture. This, coupled with the quality variation seen in many chrome steel wire, makes the determination of a single heat treatment procedure for all virtually impossible

CHAPTER IV

MATERIALS and METHODS

Standard wire testing method

The International Organization for Standardization reported the mechanical testing of metals in international standard ISO 15841:2006. Wire are classified on the basis of their elastic behaviour [19]

- a) Type 1 wires: wire displaying linear elastic behaviour during unloading at temperatures up to 50° c
- b) Type 2 wires: wire not displaying linear elastic behaviour during unloading at temperatures up to 50° c

Stainless steel is type 2, the stainless wire was tested according to the method below:

Materials

1. The stainless steel wire samples are divided into 3 groups :

Group 1 : Commercial orthodontic wire.

Group 2 : Commercial general purpose wire.

Group 3 : Drawn wire (from commercial general purpose wire).

Group 1 was purchased from the distributors in Thailand. Group 2 was ordered from Small Part Inc, USA. All samples with different diameters are shown in Table 1.

Code	Diameter(mm)	Description (company)		
COM1	0.5	Commercial orthodontic wire(3M)		
COM1HT	0.5	Heat-treated commercial orthodontic wire(3M)		
COM2	0.5	Commercial orthodontic wire(Accord)		
COM3	0.5	Commercial orthodontic wire(Highland)		
GEN1	0.5	Commercial general purpose wire(Small Parts Inc)		
GEN2	0.55	Commercial general purpose wire(Small Parts Inc)		
GEN3	0.6	Commercial general purpose wire(Small Parts Inc)		
GEN4	0.725	Commercial general purpose wire(Small Parts Inc)		
GEN2A	0.55	Annealing of Commercial general purpose wire(Small Parts Inc)		
GEN3A	0.6	Annealing of Commercial general purpose wire(Small Parts Inc)		
GEN4A	0.725	Annealing of Commercial general purpose wire(Small Parts Inc)		
DRAWN1	0.5	The wire that has been drawn with reduction ratio of 20%		
DRAWN2	0.5	The wire that has been drawn with reduction ratio of 30%		
DRAWN3	0.5	The wire that has been drawn with reduction ratio of 50%		

Table 1.	Stainless ste	el wire used	l in the	experiment
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2. Wire drawing die

Made from tungsten carbide standard type W 103 (JIS B4111). It had die bearing 1mm., Approch angle 12. Internal diameter 0.5 and 0.6 mm.


Figure 5. Drawing die

3. Oil lubricant (synthetic)

It used for lubricate drawing process ,ISO CUT570-A(FOCUS)

Study Design

There are 3 sections of experiment that are designed to measure and compare mechanical, physical and chemical properties which are diameter, composition, ultimate tensile strength, yield strength, stiffness, %elongation, modulus of elasticity, surface hardness, cross sectional hardness, surface roughness and corrosion. Six samples in each group were used for measurement in all mechanical properties.

		Stainless steel wire (No. of sample)												
	Orthodontic			Gen	General purpose wire				Drawn wire					
	wire	e												
	С	С	С	С	G	G	G	G	G	G	G	D	D	DR
	0	0	0	0	Е	Е	Е	Ε	Е	Е	Е	R	R	AW
	Μ	Μ	Μ	Μ	Ν	Ν	Ν	Ν	Ν	Ν	Ν	Α	Α	N3
	1	1	2	3	1	2	3	4	2	3	4	W	W	
		H							Α	Α	Α	Ν	Ν	
		Т										1	2	
Diameter	6	6	6	6	6	6	6	6	6	6	6	6	6	6
%Elongation	6	6	6	6	6	6	6	6	6	6	6	6	6	6
Ultimate	6	6	6	6	6	6	6	6	6	6	6	6	6	6
tensile														
strength														
Siffness	6	6	6	6	6	6	6	6	6	6	6	6	6	6
Yield	6	6	6	6	6	6	6	6	6	6	6	6	6	6
strength														
Modulus of	6	6	6	6	6	6	6	6	6	6	6	6	6	6
elasticity														
Surface	6	6	6	6	6	6	6	6	6	6	6	6	6	6
hardness														
Cross	6	6	6	6	6	6	6	6	6	6	6	6	6	6
sectional														
hardness														
Corrosion	4	4	4	4	4	-	I	-	-	I	-	4	4	4

Table 2. Study design in comparing physical, mechanical and chemical properties

Methods and procedures

First section was designed to measure and compare mechanical, physical and chemical properties including diameter, composition, ultimate tensile strength, yield strength, %elongation, modulus of elasticity, stiffness, surface hardness, cross sectional hardness, surface roughness and corrosion of commercial orthodontic wire and commercial general purpose wire.

1 Physical properties

1.1 Diameter

Six specimens of a single product from one batch is procured for test. Measurement used by micrometers follow ISO 15841:2006(E)



Figure 6. Micrometer

1.2 Surface roughness (Surface characteristics)

Six specimens of a single product from one batch is procured for test. According to ISO 15841:2006(E), Surface characteristics of each of the specimens of wires from 3 groups (Orthodontic wire, General purpose wire, Drawn wire) were studied by a scanning electron microscope (SEM) (JSM-5410LV JEOL LTD,Tokyo,Japan). A one cm-long specimen of each alloy wire was mounted on studs, which were later placed in the vacuum chamber of the SEM. The accelerating voltage, angle of fit, and the aperture was adjusted to optimize the quality of the micrograph. The surface was scanned and viewed on the monitor at different magnifications and representative micrographs (X100) of each alloy were obtained.



Figure 7. Scanning electron microscope: JSM-5410LV JEOL LTD, Tokyo, Japan

1.3 Composition

Wire specimens were examined using scanning electron microscopy and energydispersive X-ray microanalysis (SEM-EDS: JSM-5410LV JEOL LTD, Tokyo, Japan) to assess the elemental composition of the wires. For this purpose, wire segments were bonded to aluminium stabs, vacuum coated with a thin layer of conductive carbon, and examined under an SEM unit. Spectra were obtained at two randomly selected regions on the surface of the wires under the following conditions: 20 kV accelerating voltage, 50 μ A beam current, 500x original magnification and 120 seconds acquisition time.



Figure 8. Energy-dispersive X-ray microanalysis (SEM-EDS: JSM-5410LV JEOL LTD, Tokyo, Japan)

1.4 Grain structure

The specimens from drawn wire group were subjected to metallurgical analysis after polishing and etching to evaluate the morphology and structure of alloy surfaces. Cross-sectional surfaces of the specimens were embedded in epoxy resin using Polyvinyl chloride pipes as a mold and were polished with 600-1,200 grit size SiC papers followed by 1.0 and 0.3 mm alumina paste. Specimens were further etched with an etching solution of concentrated hydrofluoric acid: nitric acid: and distilled water at 1:4:5 volume ratios to reveal the grain structure of alloys. Drawn wire group were studied under scanning electron microscope for analysis of the grain size.

2 Mechanical properties

2.1 Hardness testing

This study investigates hardness at surface and cross sectional areas. The surface hardness was measured by using 6 pieces of wire in each specimen mouted on plate. By stripping of double-sided tape between plate and wire for prevent rolling upon indentation, the position of the lateral aspects of the wire, as viewed under low power objective, was recorded and the central point of the wire was calculated. Before indenting, the focus was adjusted under high power objective to ensure the correct distance between the diamond tip and the sample to be tested. The test weight provided a loading force of 50 gram for a set period of 15 seconds. In cross sectional hardness test, cross section area was separated in 3 points, 1 reference is center of wire, 2 reference is 0.1 mm away from point 1, 3 reference is 0.2 mm away from point1. By using loading force of 50 gram for a set period of 15 seconds same test as surface hardness test was performed. The hardness tester was precalibrated to provide an indentation time of 15 seconds, which is in accordance with the BSI guidelines "5411"[20]. The diagonals (dl and d,) of the diamond indent were measured under the high power objective. The Vickers hardness number (HV) was calculated by first determining the arithmetic mean of the two measured diagonals, then using the following formula:

HV = 189 x F x 103

D2

where F = the force used to indent (in newtons), 189 = Vickers constant, and D = the arithmetic mean of the two diagonals in micrometers.

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Figure 9. Setup for cross section hardness



Figure 10. Point1,2,3 in cross sectional hardness test

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Figure 11. Surface hardness test

2.2 Tensile test

A standard tensile test using each of specimen from 3 groups(orthodontic wire, general purpose wire, drawn wire) were performed in an Instron Universal Testing Machine (Model No. 1195, Instron Corporation, Canton, Mass, USA). A full-scale load of 1000 N was set in the machine with a crosshead speed of 2 mm/minute. The span of the wire between crossheads was standardized as 40 mm. and attach extensometer was used for measured %elongation. The load taken to break the wire divided by the cross-sectional area of the wire gave the value for ultimate tensile strength. The load deflection data obtained from the tensile testing were plotted as stress-strain curves from which the Modulus of elasticity as well as 0.2% offset yield strength were calculated.



Figure 12. Instron Universal Testing Machine

2.3 Three-point bending test

All groups of testing were 6 samples/specimen. The apparatus used for the three-point bending test is a further development of the device described by ISO 15841:2006(E). In this study, the wire specimens were cut to 30 mm. The specimens were subjected to a symmetrical three-point bending test. A span of wire 10 mm. between supports was used. Deflection was carried out with a centrally placed indenter. The supports and indenter had an edge curvature between 0.05 mm. and 0.13 mm. The mid-span deflection rate was 0.5 mm./min. The three-point bending test was carried out under room temperature $23\pm2^{\circ}$ C. The wires were deflected to 2 mm. and reading.

The load cell registered the force placed on the wire specimen and transmitted this value to a computer. The stiffness was derived for all test wires from the slopes of the load-deflection plot.

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Figure 13. Three-point bending test

3. Chemical properties

3.1 Corrosion test

Following, International standard ISO 10271 [21], corrosion test was done. By using electrochemical test; a potentiostat (Solartron 1285) was used to perform the linear polarization test. Stainless steel wires (Commercial orthodontic wire group, General purpose wire, Drawn wire group) were used as working electrodes. A saturated calomel electrode and platinum sheet were used as the reference electrode and counter electrode, respectively. Electrolyte was used as the corrosion test, which consisted of dissolving 9 gram of sodium chloride in approximately 950 ml water, Adjusted to pH 7.4, by using 1% lactic acid or 4% sodium hydroxide. Finally, dilute with water to 1000 ml. The electrolyte was deaerated with nitrogen gas for one hour before the specimen was dipped into the electrolyte for the following corrosion test. The linear polarization curves of the test specimens were measured with a scan rate of 0.1 mV/second after dipping the specimen into the test electrolyte for two hours. The corrosion potential, is defined as the slope of the potential vs the current density in the linear polarization curves. The sample size for the corrosion test of each wire was 4.



Figure 14. pH Meter



Figure 15. Potentiostat (Solartron 1285)

<u>Second section</u> Commercial general purpose wire of 3 diameters (0.55,0.6,0.725 mm.) was annealed at 1040° C, for 1 minute and slow cooled down to room temperature. Then, oxide that was presented on the surface of the wire was eliminated with acid solution. It contains hydrofruolic, nitric and water in composition ratio of 1:4:5. After removing of oxide all wires were proceeded for drawing.

Diameters of 0.55, 0.6, 0.725 mm. were drawn through the die made of tungsten carbide in order to their final diameter of 0.5mm. Consequently, the reduction ratios of cross sectional area were 20 and 30%, 50%, respectively. The die approach angle was 12° . Drawing speed was constant at 0.41 mm/s. Reduction ratios were 20% and

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30% drawn via single die approach but was 50% via double die (0.5 and 0.6 mm.) approach.



Figure16. Wire drawing process

Third section Measurement and evaluation mechanical, physical and corrosion properties included diameter, composition, ultimate tensile strength, yield strength, %elongation, modulus of elasticity, stiffness, surface hardness, cross sectional hardness, surface roughness and corrosion of drawn wire in each %reduction ratio. The method was similar to the first section. Finally, the results were compared among all groups of wires.



Figure 17. Diagram of wire drawing process

For comparison purpose, equivalent units of force and distance are listed below:

1 Oz = 28.35 grams
 1 Newton = 101.98 grams force
 1 inch = 25.4 mm.

Research Hypothesis

Physical property

Ho : There is no difference in means diameter among different group of wire (COM1, COM1HT, COM2, COM3, GEN1, DRAWN1, DRAWN2, DRAWN3).

Mechanical properties

Ho : There is no difference in surface hardness among different group of wire (COM1, COM1HT, COM2, COM3, GEN1, DRAWN1, DRAWN2, DRAWN3).

Ho : There is no difference in cross sectional hardness among different group of wire (COM1, COM1HT, COM2, COM3, GEN1, DRAWN1, DRAWN2, DRAWN3).
Ho : There is no difference in ultimate tensile strength among different group of wire

(COM1, COM1HT, COM2, COM3, GEN1, DRAWN1, DRAWN2, DRAWN3).

Ho: There is no difference in yield strength among different group of wire (COM1, COM1HT, COM2, COM3, GEN1, DRAWN1, DRAWN2, DRAWN3).

Ho : There is no difference in stiffness among different group of wire (COM1, COM1HT, COM2, COM3, GEN1, DRAWN1, DRAWN2, DRAWN3).

Ho: There is no difference in modulus of elasticity among different group of wire (COM1, COM1HT, COM2, COM3, GEN1, DRAWN1, DRAWN2, DRAWN3).

Ho: There is no difference in %elongation among different group of wire (COM1, COM1HT, COM2, COM3, GEN1, DRAWN1, DRAWN2, DRAWN3).

Statistical Analysis

Statistical analysis was performed by using Statistical Package for the Social Science(SPSS) for windows version 11.4. Results were presented as mean±SD. Kolmogorov-Smirnov was used to check normality of data in each group and One Way ANOVA and multiple comparison (Turkey or Dunnett's T3), were used to compare the mean diameter, surface hardness, cross sectional hardness, yield strength, ultimate tensile strength, modulus of elasticity, %elongation among different group

(COM1, COM1HT, COM2, COM3, GEN1, DRAWN1, DRAWN2, DRAWN3). If variance was not equal in each group then Welch test was used instead. The level of statistical significant difference was considered at P<0.05.

CHAPTER V RESULTS

Part I: Physical Properties

Composition

Wires in Commercial orthodontic wire group and General purpose wire group were randomly selected to test whether the composition of wire was truly the 18:8 stainless steel. Seven ions were considered, these included carbon, manganese, sulfur, chromium, iron, phosphorus and nickel. Selected wires were GEN1, GEN2, COM1 and COM1HT. The results confirmed that these wires were genuine stainless type 304.

	C	Mg	Р	S	Cr	Fe	Ni
COM1	1.45	0.12	0.06	0.05	19.74	69.90	8.68
COM1HT	2.31	0.09	0.14	0.03	18.68	71.00	7.74
GEN1	1.08	0.01	0.04	0.01	19.38	72.38	7.09
GEN2	1.30	0.02	0.01	0.06	19.53	71.47	7.62

Table 3. % of composition in each wire

Diameter

Tables 4,5 and Figure 18 showed means, standard deviations and multiple comparison of diameter of all sample groups. In terms of Diameter, significant difference (p<0.05) had been found when using ANOVA test. However, diverse results occurred when using Multiple Comparison test. First, in group of Commercial orthodontic wire, all wires except for COM1 possessed the same diameter. Second, no difference of Diameter had been found in Drawn wire group no matter what reduction ratios of cross sectional area were used. Finally, Diameter of General purpose wire (GEN1) was not different from wires in Drawn wire group and COM1.

Wire type $(n = 6)$	Mean	Std. Deviation
COM1	.504	.001
COM1HT	.511	.001
COM2	.510	.003
COM3	.510	.003
GEN1	.502	.003
GEN2	.551	.001
GEN3	.605	.001
GEN4	.715	.001
DRAWN1	.500	.002
DRAWN2	.503	.002
DRAWN3	.502	.004

 Table 4. Means and standard deviations of diameter (mm.)

Wire type	Wire type	P-value
	COM1HT	.003*
	COM2	.012*
	COM3	.012*
COM1	GEN1	.974
	DRAWN1	.286
	DRAWN2	.997
	DRAWN3	.901
	COM1	.003*
	COM2	1.000
	COM3	1.000
COM1HT	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.012*
	COM1HT	1.000
	COM3	1.000
COM2	GEN1	.001*
	DRAWN1	.000*
	DRAWN2	.002*
	DRAWN3	.000*
	COM1	.012*
	COM1HT	1.000
	COM2	1.000
COM3	GEN1	.001*
	DRAWN1	.000*
	DRAWN2	.002*
	DRAWN3	.000*

 Table 5.
 Multiple comparison of diameter by Tukey test.

* The mean difference is significant at the 0.05 level

Wire type Wire type P-value COM1 .974 COM1HT 000*

* The mean difference is significant at the 0.05 level

	COM1HT	.000*
	COM2	.001*
GEN1	COM3	.001*
	DRAWN1	.861
	DRAWN2	1.000
	DRAWN3	1.000
	COM1	.286
	COM1HT	.000*
	COM2	.000*
DRAWN1	COM3	.000*
	GEN1	.861
	DRAWN2	.703
	DRAWN3	.957
	COM1	.997
	COM1HT	.000*
	COM2	.002*
DRAWN2	COM3	.002*
	GEN1	1.000
	DRAWN1	.703
	DRAWN3	.999
	COM1	.901
	COM1HT	.000*
	COM2	.000*
DRAWN3	COM3	.000*
	GEN1	1.000
	DRAWN1	.957
	DRAWN2	.999



Figure 18. Bar graph of diameter

Surface roughness

In terms of Surface Roughness, results from using SEM with view x100 revealed that wires in Commercial orthodontic wire group possessed the smoothest surface especially COM1, followed by GEN1, while the roughest surface was found in Drawn wire group. In drawn wire group, wire with 50% reduction ratio (DRAWN3) had smoother surface than those with reduction ratios of 20% and 30%. Illustrations can be seen in Figures 19-32.

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Figure 19. Commercial orthodontic wire : COM1 (SEM X100)



Figure 20. Commercial orthodontic wire : COM1HT (SEM X100)



Figure 21. Commercial orthodontic wire : COM2 (SEM X100)



Figure 22. Commercial orthodontic wire : COM3 (SEM X100)



Figure 23. Commercial general purpose wire : GEN1 (SEM X100)



Figure 24. Commercial general purpose wire : GEN2 (SEM X100)

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Figure 25. Commercial general purpose wire : GEN3 (SEM X100)



Figure 26. Commercial general purpose wire : GEN4 (SEM X100)



Figure 27. Commercial general purpose wire after annealing at 1040 °C, 1 minute : GEN2A (SEM X100)

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Figure 29. Commercial general purpose wire after annealing at 1040 °C, 1 minute : GEN4A (SEM X100)



Figure 30. Drawn wire at reduction ratio 20% : DRAWN1 (SEM X100)

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Figure 31. Drawn wire at reduction ratio 30% : DRAWN2 (SEM X100)



Figure 32. Drawn wire at reduction ratio 50% : DRAWN3 (SEM X100)

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Grain size

From our investigation, Figures 33-35 show the microstructure in the cross sectional area of Drawn wire group at reduction ratios of 20, 30 and 50%, respectively. Wire with 50% reduction ratio had denser and smaller grain than those with reduction ratios of 20% and 30%.



Figure 33. Grain structure in cross sectional area of DRAWN1 (SEM X1000)



Figure 34. Grain structure in cross sectional area of DRAWN2 (SEM X1000)



Figure 35. Grain structure in cross sectional area of DRAWN3 (SEM X1000)

Part II: Mechanical Properties

% of Elongation

Results from Welch Test reported that the difference was statistically significant p<0.05 (0.00). % Elongation in Commercial orthodontic wire group was not different except only for COM1HT and COM2. In Drawn wire group, no difference was presented in all % of reduction ratios used in the experiment (20%, 30%, 50%). However, when compared with results from Commercial orthodontic wire group, difference in % Elongation occurred only between DRAWN3 and COM1, COM1HT, COM3. As for GEN1, there was no significant difference in Elongation with Drawn wire and Commercial orthodontic wire. The results were shown in Tables 6-7 and Figure 36.

Wire type $(n=6)$	Mean	Std. Deviation
COM1	2.026	.258
COM1HT	2.125	.235
COM2	1.604	.202
COM3	2.053	.201
GEN1	1.627	.322
GEN2	1.525	.561
GEN3	1.971	.350
GEN4	1.771	.153
GEN2 A	14.520	2.367
GEN3 A	16.335	3.736
GEN4 A	16.626	1.640
DRAWN1	8.250	3.347
DRAWN2	2.061	.506
DRAWN3	1.464	.147

Table 6. Means and standard deviations of % elongation

Wire type	Wire type	P-value
	COM1HT	1.000
	COM2	.182
	COM3	1.000
COM1	GEN1	.492
	DRAWN1	.075
	DRAWN2	1.000
	DRAWN3	.031*
	COM1	1.000
	COM2	.044*
	COM3	1.000
COM1HT	GEN1	.209
	DRAWN1	.080
	DRAWN2	1.000
	DRAWN3	.007*
	COM1	.182
	COM1HT	.044*
	COM3	.062
COM2	GEN1	1.000
	DRAWN1	.058
	DRAWN2	.675
	DRAWN3	.969
	COM1	1.000
	COM1HT	1.000
	COM2	.062
COM3	GEN1	.322
	DRAWN1	.076
	DRAWN2	1.000
	DRAWN3	.005*

Table 7. Multiple comparison of % elongation by Dunnett T3 test.

* The mean difference is significant at the 0.05 level

Wire type	Wire type	P-value
	COM1	.492
	COM1HT	.209
	COM2	1.000
GEN1	COM3	.322
	DRAWN1	.058
	DRAWN2	.825
	DRAWN3	.993
	COM1	.075
	COM1HT	.080
	COM2	.058
DRAWN1	COM3	.076
	GEN1	.058
	DRAWN2	.075
	DRAWN3	.054
	COM1	1.000
	COM1HT	1.000
	COM2	.675
DRAWN2	COM3	1.000
	GEN1	.825
	DRAWN1	.075
	DRAWN3	.354
	COM1	.031*
	COM1HT	.007*
	COM2	.969
DRAWN3	COM3	.005*
	GEN1	.993
	DRAWN1	.054
	DRAWN2	.354

 Table 7.
 Multiple comparison of % elongation by Dunnett T3 test. (cont.)

* The mean difference is significant at the 0.05 level



Figure 36. Bar graph of % elongation

Ultimate tensile strength

Results from Welch Test demonstrated that the difference was statistically significant (p<0.05). It seemed that all groups of Commercial orthodontic wire, General purpose wire (GEN1) and Drawn wire had different Ultimate tensile strength in all pairs tested. According to Tables 8-9 and Figure 37, Drawn wire with 20% and 30% reduction ratio had lower Ultimate tensile strength than those of Commercial orthodontic wire and General purpose wire. Also, the study further reported that higher Ultimate tensile strength was found in Drawn wire with 50% reduction ratio when compared with 20% and 30% reduction ratio.

Wire type (n=6)	Mean	Std. Deviation
COM1	2088	13.42
COM1HT	2599	21.92
COM2	2716	32.83
COM3	2239	1.39
GEN1	2120	1.17
GEN2	2023	4.26
GEN3	2113	5.35
GEN4	1882	5.25
GEN2 A	744	18.21
GEN3 A	709	14.10
GEN4 A	819	58.95
DRAWN1	1046	13.10
DRAWN2	1241	17.56
DRAWN3	1755	56.86

Table 8. Means and standard deviations of ultimate tensile strength (MPa)

Wire type	Wire type	P-value
	COM1HT	.000*
	COM2	.000*
	COM3	.000*
COM1	GEN1	.027*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.000*
	COM2	.001*
	COM3	.000*
COM1HT	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.001*
	COM3	.000*
COM2	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
COM3	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*

Table 9. Multiple comparison of UTS by Dunnett T3 test.

*The mean difference is significant at the 0.05 level

Wire type	Wire type	P-value
	COM1	.027*
	COM1HT	.000*
	COM2	.000*
GEN1	COM3	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN1	COM3	.000*
	GEN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN2	COM3	.000*
	GEN1	.000*
	DRAWN1	.000*
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN3	COM3	.000*
	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*

Table 9. Multiple comparison of UTS by Dunnett T3 test. (cont.)

* The mean difference is significant at the 0.05 level



Figure 37. Bar graph of ultimate tensile strength.

Yield strength

From Tables 10-11 and Figure 38, Commercial orthodontic wire and General purpose wire (GEN1) had superior Yield strength than Drawn wire with 20% and 30% reduction ratio. When compared wires within Drawn wire group, the more % of reduction ratio, the more Yield strength was noticeably found. In addition, results from ANOVA test revealed that the difference was statistically significant p<0.05. Moreover, difference Yield strength was also found across Commercial orthodontic wire group. As for Drawn wire group, wires with 20% and 30% reduction ratio did not contain much difference in Yield strength from each other, but much higher in Yield strength was reported in wire with 50% reduction ratio.

Wire type (n=6)	Mean	Std. Deviation
COM1	1651	49.49
COM1HT	2079	96.87
COM2	2610	59.72
COM3	1784	147.09
GEN1	1810	202.33
GEN2	1749	104.20
GEN3	1717	90.53
GEN4	1614	130.83
GEN2 A	295	18.71
GEN3 A	247	19.24
GEN4 A	368	14.95
DRAWN1	787	33.77
DRAWN2	937	49.06
DRAWN3	1648	61.01

 Table 10.
 Means and standard deviations of yield strength (MPa).

Wire type	Wire type	P-value
	COM1HT	.000*
	COM2	.000*
	COM3	.352
COM1	GEN1	.161
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	1.000
	COM1	.000*
	COM2	.000*
	COM3	.000*
COM1HT	GEN1	.001*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.000*
	COM3	.000*
COM2	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.352
	COM1HT	.000*
	COM2	.000*
COM3	GEN1	1.000
	DRAWN1	*000
	DRAWN2	.000*
	DRAWN3	.327

 Table 11.
 Multiple comparison of yield strength by Tukey test.

*The mean difference is significant at the 0.05 level

Wire type	Wire type	P-value
	COM1	.161
	COM1HT	.001*
	COM2	.000*
GEN1	COM3	1.000
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.146
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN1	COM3	.000*
	GEN1	.000*
	DRAWN2	.221
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN2	COM3	.000*
	GEN1	.000*
	DRAWN1	.221
	DRAWN3	.000*
	COM1	1.000
	COM1HT	.000*
	COM2	.000*
DRAWN3	COM3	.327
	GEN1	.146
	DRAWN1	.000*
	DRAWN2	.000*

 Table 11.
 Multiple comparison of yield strength by Tukey. (cont.)

* The mean difference is significant at the 0.05 level


Figure 38. Bar graph of yield strength

Stiffness

Graph of three point bending test and slope of graph represented stiffness. Wires in Commercial orthodontic wire group, GEN1 and drawn wire group had similar stiffness. Furthermore, when compared within size, it was found that large diameter wire had more stiffness than that of small diameter. Annealing can reduce stiffness when compared to as-received wires. Results are shown in Table 12 and Figure 39.

Wire type (n=6)	Mean	Std. Deviation
COM1	20000	0
COM1HT	20000	0
COM2	25000	0
COM3	20000	0
GEN1	21428	0
GEN2	27777	0
GEN3	40000	0
GEN4	83333	0
GEN2 A	20000	0
GEN3 A	32500	0
GEN4 A	40000	0
DRAWN1	20000	0
DRAWN2	20000	0
DRAWN3	17142	0

Table 12. Means and standard deviations of stiffness (N/m).



Figure 39. Bar graph of stiffness

Young's modulus of elasticity

No statistically difference was found in all 3 groups of wires using ANOVA test p>0.05 (0.093) as shown in Tables 9-10 and Figure 40.

Wire type (n=6) Mean Std. Deviation COM1 196 38.38 COM1HT 206 16.48 40.02 COM2 239 COM3 205 28.00 GEN1 187 12.62 GEN2 227 43.32 GEN3 214 25.92 GEN4 214 19.80 GEN2 A 172 56.18 GEN3 A 137 33.37 GEN4 A 162 39.50 36.29 DRAWN1 196 DRAWN2 211 37.87 DRAWN3 180 32.54

 Table 13.
 Means and standard deviations of Young' modulus of elasticity (GPa).

Wire type	Wire type	P-value
	COM1HT	.999
	COM2	.295
	COM3	1.000
COM1	GEN1	1.000
	DRAWN1	1.000
	DRAWN2	.990
	DRAWN3	.988
	COM1	.999
	COM2	.610
	COM3	1.000
COM1HT	GEN1	.967
	DRAWN1	1.000
	DRAWN2	1.000
	DRAWN3	.860
	COM1	.295
	COM1HT	.610
	COM3	.593
COM2	GEN1	.111
	DRAWN1	.301
	DRAWN2	.793
	DRAWN3	.049
	COM1	1.000
	COM1HT	1.000
	COM2	.593
COM3	GEN1	.971
	DRAWN1	1.000
	DRAWN2	1.000
	DRAWN3	.872

Table 14. Multiple comparison of modulus of elasticity by Tukey test.

*The mean difference is significant at the 0.05 level

Wire type	Wire type	P-value
	COM1	1.000
	COM1HT	.967
	COM2	.111
GEN1	COM3	.971
	DRAWN1	1.000
	DRAWN2	.881
	DRAWN3	1.000
	COM1	1.000
	COM1HT	1.000
	COM2	.301
DRAWN1	COM3	1.000
	GEN1	1.000
	DRAWN2	.991
	DRAWN3	.987
	COM1	.990
	COM1HT	1.000
	COM2	.793
DRAWN2	COM3	1.000
	GEN1	.881
	DRAWN1	.991
	DRAWN3	.698
	COM1	.988
	COM1HT	.860
	COM2	.049
DRAWN3	COM3	.872
	GEN1	1.000
	DRAWN1	.987
	DRAWN2	.698

 Table 14.
 Multiple comparison of modulus of elasticity by Tukey. (cont.)

* The mean difference is significant at the 0.05 level



Figure 40. Bar graph of Young's modulus of elasticity

Surface vicker hardness

According to Tables 15-16 and Figure 41, Surface vicker hardness of wires in Commercial orthodontic wire group and General purpose wire group were higher than those of Drawn wire with 20% and 30% reduction ratio. Furthermore, drawn wire with 50% reduction ratio seemed to have higher Surface vicker hardness level than wires with 20% and 30% reduction ratio. From statistics test (Welch Test), no difference in Surface vicker hardness level were seen between GEN1 and all wires in Commercial orthodontic wire group together with drawn wire with 50% reduction ratio.

Wire type (n=6)	Mean	Std. Deviation
COM1	423.06	14.37
COM1HT	412.97	33.68
COM2	493.59	25.84
COM3	413.92	29.53
GEN1	519.50	64.76
GEN2	520.15	12.81
GEN3	525.41	34.20
GEN4	505.07	14.63
GEN2 A	197.39	11.01
GEN3 A	178.05	13.08
GEN4 A	169.58	16.24
DRAWN1	355.70	13.79
DRAWN2	379.60	26.50
DRAWN3	402.14	14.56

 Table 15.
 Means and standard deviations of surface hardness (HV)

Wire type	Wire type	P-value
	COM1HT	1.000
	COM2	.008*
	COM3	1.000
COM1	GEN1	.167
	DRAWN1	.000*
	DRAWN2	.127
	DRAWN3	.416
	COM1	1.000
	COM2	.022*
	COM3	1.000
COM1HT	GEN1	.123
	DRAWN1	.102
	DRAWN2	.758
	DRAWN3	1.000
	COM1	.008*
	COM1HT	.022*
	COM3	.012*
COM2	GEN1	.999
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.001*
	COM1	1.000
	COM1HT	1.000
	COM2	.012*
COM3	GEN1	.124
	DRAWN1	.052
	DRAWN2	.634
	DRAWN3	1.000

Table 16. Multiple comparison of surface hardness by Dunnett T3 test.

*The mean difference is significant at the 0.05 level

Wire type	Wire type	P-value
	COM1	.167
	COM1HT	.123
	COM2	.999
GEN1	COM3	.124
	DRAWN1	.019*
	DRAWN2	.033*
	DRAWN3	.080
	COM1	.000*
	COM1HT	.102
	COM2	.000*
DRAWN1	COM3	.052
	GEN1	.019*
	DRAWN2	.725
	DRAWN3	.005*
	COM1	.127
	COM1HT	.758
	COM2	.000*
DRAWN2	COM3	.634
	GEN1	.033*
	DRAWN1	.725
	DRAWN3	.796
	COM1	.416
	COM1HT	1.000
	COM2	.001*
DRAWN3	COM3	1.000
	GEN1	.080
	DRAWN1	.005*
	DRAWN2	.796

 Table 16.
 Multiple comparison of surface hardness by Dunnett T3 test. (cont.)

* The mean difference is significant at the 0.05 level



Figure 41. Bar graph of surface hardness

Cross section vicker hardness

From Tables 17-22 and Figure 42, results of this measurement on 3 points of wire were similar in that high level of Cross section vicker hardness was seen in all 3 points of wires in Commercial orthodontic wire group and General purpose wire group (GEN1) than in Drawn wire with 20% and 30% reduction ratio. In addition, higher level of hardness was found in wire with 50% reduction ratio when compared with drawn wire with 20% and 30% reduction ratio. Outcome of ANOVA and Welch tests in 3 points of wires reported statistically significant difference in every point, point1 p<0.05 (.000), point2 p<0.05 (.000), and point3 p<.005 (.000)

Wire type (n=6)	Mean	Std. Deviation
COM1	498.84	6.74
COM1HT	532.41	11.00
COM2	619.29	11.23
COM3	456.25	14.37
GEN1	502.18	9.26
GEN2	538.04	16.49
GEN3	501.48	13.87
GEN4	484.99	11.26
GEN2 A	204.49	8.36
GEN3 A	202.82	4.06
GEN4 A	197.68	8.07
DRAWN1	288.06	13.86
DRAWN2	357.66	9.96
DRAWN3	510.93	25.25

 Table 17.
 Means and standard deviations of cross section hardness in point 1 (HV).

 Table 18.
 Means and standard deviations of cross section hardness in point 2 (HV).

Wire type (n=6)	Mean	Std. Deviation
COM1	541.51	9.55
COM1HT	597.21	7.54
COM2	677.48	15.87
COM3	499.72	9.62
GEN1	533.32	11.34
GEN2	548.01	7.99
GEN3	525.22	8.49
GEN4	498.88	19.29
GEN2 A	207.27	8.84
GEN3 A	195.27	10.43
GEN4 A	199.10	7.32
DRAWN1	320.52	16.23
DRAWN2	373.03	11.08
DRAWN3	527.67	35.81

Wire type (n=6)	Mean	Std. Deviation
COM1	498.11	10.52
COM1HT	551.53	26.01
COM2	686.70	18.36
COM3	526.29	9.43
GEN1	504.17	31.35
GEN2	520.81	7.40
GEN3	535.11	10.11
GEN4	496.37	14.75
GEN2 A	207.03	9.92
GEN3 A	198.64	9.33
GEN4 A	205.42	8.59
DRAWN1	341.75	16.88
DRAWN2	392.12	4.76
DRAWN3	519.13	20.35

Table 19. Means and standard deviations of cross section hardness in point 3 (HV).

Wire type	Wire type	P-value
	COM1HT	.003*
	COM2	.000*
	COM3	.000*
COM1	GEN1	1.000
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.791
	COM1	.003*
	COM2	.000*
	COM3	.000*
COM1HT	GEN1	.010*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.151
	COM1	.000*
	COM1HT	.000*
	COM3	.000*
COM2	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
COM3	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*

Table 20. Multiple comparison of hardness, point 1 by Tukey test.

*The mean difference is significant at the 0.05 level

Wire type	Wire type	P-value
	COM1	1.000
	COM1HT	.010*
	COM2	.000*
GEN1	COM3	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.953
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN1	COM3	.000*
	GEN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN2	COM3	.000*
	GEN1	.000*
	DRAWN1	.000*
	DRAWN3	.000*
	COM1	.791
	COM1HT	.151
	COM2	.000*
DRAWN3	COM3	.000*
	GEN1	.953
	DRAWN1	.000*
	DRAWN2	.000*

Table 20. Multiple comparison of hardness, point 1 by Tukey test (cont.)

Wire type	Wire type	P-value
	COM1HT	.000*
	COM2	.000*
	COM3	.000*
COM1	GEN1	.973
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.999
	COM1	.000*
	COM2	.000*
	COM3	.000*
COM1HT	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.060
	COM1	.000*
	COM1HT	.000*
	COM3	.000*
COM2	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.001*
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
COM3	GEN1	.006*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.777

Table 21. Multiple comparison of hardness, point 2 by Dunnett T3 test.

*The mean difference is significant at the 0.05 level

Wire type	Wire type	P-value
	COM1	.973
	COM1HT	.000*
	COM2	.000*
GEN1	COM3	.006*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	1.000
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN1	COM3	.000*
	GEN1	.000*
	DRAWN2	.002*
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN2	COM3	.000*
	GEN1	.000*
	DRAWN1	.002*
	DRAWN3	.001*
	COM1	.999
	COM1HT	.060
	COM2	.001*
DRAWN3	COM3	.777
	GEN1	1.000
	DRAWN1	.000*
	DRAWN2	.001*

Table 21. Multiple comparison of hardness, point 2 by Dunnett T3 test (cont.)

* The mean difference is significant at the 0.05 level

Wire type	Wire type	P-value
	COM1HT	.000*
	COM2	.000*
	COM3	.203
COM1	GEN1	.999
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.555
	COM1	.000*
	COM2	.000*
	COM3	.325
COM1HT	GEN1	.003*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.092
	COM1	.000*
	COM1HT	.000*
	COM3	.000*
COM2	GEN1	.000*
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.000*
	COM1	.203
	COM1HT	.325
	COM2	.000*
COM3	GEN1	.491
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.998

 Table 22.
 Multiple comparison of hardness, point 3 by Tukey test.

*The mean difference is significant at the 0.05 level

Wire type	Wire type	P-value
	COM1	.999
	COM1HT	.003*
	COM2	.000*
GEN1	COM3	.491
	DRAWN1	.000*
	DRAWN2	.000*
	DRAWN3	.871
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN1	COM3	.000*
	GEN1	.000*
	DRAWN2	.001*
	DRAWN3	.000*
	COM1	.000*
	COM1HT	.000*
	COM2	.000*
DRAWN2	COM3	.000*
	GEN1	.000*
	DRAWN1	.001*
	DRAWN3	.000*
	COM1	.555
	COM1HT	.092
	COM2	.000*
DRAWN3	COM3	.998
	GEN1	.871
	DRAWN1	.000*
	DRAWN2	.000*

Table 22. Multiple comparison of hardness, point 3 by Tukey test. (cont.)

* The mean difference is significant at the 0.05 level

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Figure 42. Bar graph show cross section hardness in point 1,2,3

Part III: Chemical property

Corrosion test

According to Figures 44-51, commercial orthodontic wires and general purpose wire generally had longer distance of slope when compared to the slope of drawn wire group. The results can be interpreted that commercial orthodontic and general purpose wire had better corrosion resistance. This can be seen in Figure 43 which showed that corrosion (rust) was found only in drawn wire group.



Figure 43. Corrosion in drawn wire group



Figure 44. Corrosion graph of COM1



Figure 45. Corrosion graph of COM1HT



Figure 46. Corrosion graph of COM2



Figure 47. Corrosion graph of COM3



Figure 48. Corrosion graph of GEN1



Figure 49. Corrosion graph of DRAWN1



Figure 50. Corrosion graph of DRAWN2



Figure 51. Corrosion graph of DRAWN3

CHAPTER VI DISCUSSION

From this study, the overall results revealed that Commercial orthodontic wires possess superior quality in relation to physical and chemical properties than Drawn wires. According to literature reviews, orthodontic stainless steel wires with high strength and low % of elongation are suitable for orthodontic use, especially during canine retraction phase as such wires are bending resistant during the movement of canines. The composition of all wires indicated that all of them were 18:8 stainless steel wire (type 304) even though %carbon atom showed higher than the standard of Type 304 (more than 0.08%). This result may be due to error from EDS method that is sensitive to carbon atom assessment.

With regard to the diameter, the result showed that one commercial orthodontic wire (COM1) had significantly different diameter when comparing among the commercial wire group. This is consistent with many studies [22-24] which states that the dimensions of materials such as archwires and brackets might be smaller than or exceed the dimensions stated by the manufacturers. When focusing on the diameter of the Drawn wire group, it is noticable that their diameters were relatively equal. This result implies that wire reduction process in this study is effective.

In terms of surface roughness, commercial orthodontic wires were relatively smooth especially COM1 and COM1HT. Surface evaluation of an archwire alloy is important because of its influence on working characteristics as well as corrosion potential. The implication of wire surface roughness on friction during sliding has not been unequivocally defined. The vast majority of the in vitro studies dealing with this issue have shown that friction increases with increased roughness of the wire and bracket surfaces.[25,26,27,28] Those studies indicated that, in general, β -Ti and NiTi wires and ceramic brackets present increased friction due to their roughened surfaces during manufacture. However, Kusy and Whitley [29] has proposed that friction is independent of wire roughness. It should be kept in mind that the results of those in vitro studies may not be applied to oral environment where biofilm and calcified

regions are included. With this regard, the smoothness of wire can reduce adhesion of plaque.

Also, the finding on chemical properties showed no corrosion in Commercial orthodontic wires and General purpose wire when compared with Drawn wire. This result may be explained that commercial orthodontic wire has smoother surface than other wire groups. By using SEM at X100, it was shown that Drawn wire in 20,30% reduction ratio had crevice surface due to acid etching process after annealing to eliminate oxide on wire surface. Though Drawn wire in 50% reduction ratio had less crevice surface than other drawn wires group but corrosion effect can still occurred. Possible cause can arise from the scratch on surface which may be the effect from adhesion between wire and inner of die surface or from insufficient lubricant system was insufficiency.

Crevice corrosion (or gasket corrosion) may occur in loci exposed to corrosive environments, and arises from differences in metal ion or oxygen concentration between the crevice and its vicinity. Severe disintegration on the surface is evident, with the formation of craters, deep fissures, and excessive pores. [30]

Because the release of nickel ions from orthodontic alloys is a clinical concern, general corrosion resistance of orthodontic metal has been widely investigated by many researchers [31,32,33]. Variations in manufacturing technique as well as postmanufacture finishing and polishing operation can have an effect on the corrosion behavior, the same composition can exhibit significantly different corrosion behavior in an in vitro artificial saliva exposure test. [33]

It is interesting to note that COM2 exhibited the highest ultimate tensile strength, yield strength, stiffness and surface roughness among the commercial group. This implies a high level of cold processing of this alloy.

Mechanical and chemical properties of General purpose wire (GEN1) are quite similar to Commercial orthodontic wire. However, in terms of physical properties, surface roughness of GEN1 was outperformed by Commercial orthodontic wire, but GEN1 still seemed to perform better than Drawn wire with every % of reduction ratio.

Outcomes of experiments on Drawn wire indicated that reduction ratios of cross sectional area at 20% and 30% did not generate enough mechanical properties to be used effectively. From the pilot study of this experiment, wire drawing at 50%

reduction ratio using only one Die caused wire broken; thus, cross sectional area of wire drawing was therefore done by using two Dies. Drawing at 50% reduction ratio increased mechanical properties of drawn wire than drawing at 20% and 30% reduction ratios. This conforms to grain structure that was denser and smaller. However, drawn wire still had worse surface roughness when compared with Commercial orthodontic wire and GEN1. This may be due to the use of acid on surface of wire in order to remove oxide from annealing process before entering drawing process. Drawn wire with 30% reduction ratio was found smoother than Drawn wire with 20% reduction ratio. Roughness of Drawn wire may also be due to adhesion between wire surface and die during wire drawing operation. This may be caused by type of lubricant used in drawing, which liquid might have less quality than solid lubricant.

In conclusion, General purpose wire may be used in orthodontics but wire surface should be improved by making it smoother. Drawn wire, 50% of reduction ratio or more seems to show an increase in mechanical properties but such drawn wire still has some weakness in surface roughness and corrosion resistance. It is suggested that to make wire drawing practical for industrial purpose, manufacturing process must be more effective and efficient. High speed and full capacity of production must be achieved together with low level of wastes and scraps from the process in order to attain economy of scale. However, there are other important factors that must be considered, for instance, speed of drawing, quality of die, number of passes, heat treatment, type of lubricant or even % of reduction ratio that has been studied in this experiment. Moreover, besides mechanical and physical properties, chemical properties as well as biocompatibility are also essential components for selection of wires used in oral cavity.

CHAPTER VII CONCLUSION

Conclusion

1. General purpose wire and commercial orthodontic wire were the same stainless steel type (Type 304) which contains approximately 18% Chromium and 8% Nickel.

2. There was no significant difference in modulus of elasticity among all types of wire used in this study.

3. Commercial orthodontic wire has smoother surfaces from polishing process during manufacturing. On the other hand, drawn wire through die drawing contained evidence of roughness from acid etch which is necessary for removing oxide after annealing process.

4. Wire drawing at 50% reduction ratio showed smoother surface than those drawn at 20% and 30% reduction ratio.

5. Ultimate tensile strength and yield strength of Commercial orthodontic wire obtained from the experiment were much higher than those of drawn wire in this research.

6. Increasing in % reduction ratio of cross sectional area in wire drawing leads to more strength of wire, and such reduction ratio should not be less than 50% in order to produce enough strength comparable to orthodontic wires.

7. Mechanical properties of General purpose wire were similar to Commercial orthodontic wire in almost all measurements except for the surface roughness that General purpose wire seemed to be outperformed by Commercial orthodontic wire according to observation results from SEM.

8. It was found that every % reduction ratio of drawn wire had corrosive reaction due to cervice on surface which caused by acid etch after annealing process.

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APPENDIX A

Type wire	Diameter	% elongation	Ultimate tensile strength	Yield strength	Young's modulus	Stiffness
COM1						
1	.505	2.193	2097.044	1591.62	168.557	20000
2	.506	1.639	2095.105	1629.82	253.105	20000
3	.505	2.232	2070.050	1697.73	188.189	20000
4	.502	2.206	2093.872	1718.95	235.204	20000
5	.504	2.126	2102.042	1655.29	173.379	20000
6	.505	1.760	2073.262	1612.84	160.717	20000
COM1HT						
1	.513	1.825	2605.129	2037.18	211.084	20000
2	.512	1.914	2627.160	2164.61	194.298	20000
3	.511	2.128	2622.420	2079.72	183.555	20000
4	.511	2.475	2578.527	1909.95	221.051	20000
5	.513	2.143	2583.088	2122.16	200.349	20000
6	.510	2.265	2580.221	2165.05	226.846	20000
COM2						
1	.510	1.752	2659.958	2546.60	200.706	25000
2	.511	1.609	2729.174	2597.53	298.202	25000
3	.514	1.416	2701.494	2699.39	213.392	25000
4	.508	1.560	2733.838	2648.46	206.076	25000
5	.515	1.378	2755.341	2546.60	274.589	25000
6	.507	1.913	2716.793	2623.00	244.921	25000
COM3						
1	.510	2.271	2240.454	1782.62	182.803	20000
2	.511	1.945	2241.464	1825.06	241.235	20000
3	.514	2.070	2238.196	1782.62	211.701	20000
4	.508	1.709	2238.958	1655.29	235.762	20000
5	.515	2.154	2238.326	1623.45	183.814	20000
6	.507	2.173	2240.834	2037.28	178.956	20000
GEN1						
1	.503	1.979	2120.290	1846.28	183.781	21428
2	.509	1.185	2121.721	1867.50	188.281	21428
3	.501	1.710	2119.602	2079.72	171.830	21428
4	.500	1.493	2122.221	1740.17	177.881	21428
5	.503	1.985	2120.403	1864.11	194.550	21428
6	.501	1.412	2119.206	1464.29	207.370	21428

Table 23. Raw data.

Type wire	Diameter	% elongation	Ultimate tensile	Yield strength	Young's modulus	Stiffness
DRAWN1			strength			
1	.504	5.134	1043.169	789.44	161.882	20000
2	.497	11.006	1045.705	753.79	190.706	20000
3	.500	7.571	1055.090	804.22	158.769	20000
4	.502	3.679	1067.069	748.70	208.128	20000
5	.499	11.957	1029.661	789.44	203.227	20000
6	.501	10.157	1038.374	840.17	257.736	20000
DRAWN2						
1	.506	1.599	1226.776	916.77	181.421	20000
2	.505	2.266	1236.050	1008.45	219.911	20000
3	.504	2.152	1227.699	916.77	262.476	20000
4	.498	2.020	1265.445	947.33	172.667	20000
5	.502	2.871	1232.443	967.70	248.850	20000
6	.505	1.461	1262.720	865.84	185.154	20000
DRAWN3						
1	.500	1.338	1677.380	1612.81	173.142	17142
2	.496	1.450	1714.110	1591.62	156.179	17142
3	.501	1.469	1829.108	1697.73	191.422	17142
4	.505	1.586	1801.733	1655.29	166.731	17142
5	.509	1.276	1774.125	1740.17	241.616	17142
6	.503	1.670	1734.301	1591.62	155.863	17142

Table 23. Raw data (cont.)

Type wire	Surface hardness	Cross section hardness (point 1)	Cross section hardness (point 2)	Cross section hardness (point 3)
COM1		¥		
1	401.40	490.71	547.94	510.44
2	423.60	500.43	553.63	500.43
3	424.01	495.53	531.38	490.71
4	435.03	495.53	536.81	505.40
5	413.27	500.43	531.38	500.43
6	441.07	510.44	547.94	481.27
COM1HT				
1	410.39	515.55	596.08	571.25
2	434.92	531.38	602.55	589.73
3	414.86	536.81	589.73	547.94
4	347.97	536.81	609.11	536.81
5	434.88	526.02	589.73	547.94
6	434.80	547.94	596.08	515.55
COM2				
1	485.36	609.11	689.04	705.25
2	541.24	636.49	689.04	689.04
3	467.45	609.11	665.75	689.04
4	502.19	629.47	681.14	705.25
5	484.28	615.79	650.87	658.24
6	481.03	615.79	689.04	673.38
COM3				
1	441.63	463.20	500.43	527.02
2	426.13	433.93	495.53	531.38
3	440.58	446.13	515.55	531.38
4	363.08	467.62	490.71	536.81
5	402.51	472.11	490.71	510.44
6	409.59	454.54	505.40	520.75
GEN1				
1	594.86	500.43	531.38	450.30
2	484.66	510.44	526.02	526.02
3	604.17	490.71	536.81	500.43
4	503.47	500.43	531.38	520.75
5	483.56	495.53	553.63	536.81
6	446.29	515.55	520.75	490.71

Table 23. Raw data (cont.)

Type wire	Surface	Cross section	Cross section	Cross section
	hardness	hardness (point 1)	hardness (point 2)	hardness (point 3)
DRAWN1		(point 1)	(point 2)	(point 3)
1	351.75	294.86	304.56	344.52
2	371.34	276.81	307.89	314.74
3	357.96	301.27	336.69	348.54
4	370.45	265.69	344.52	365.33
5	335.96	298.04	314.74	332.87
6	346.78	291.73	314.74	344.52
DRAWN2				
1	365.06	340.57	374.19	388.09
2	336.95	369.72	392.89	392.89
3	401.18	361.02	375.08	388.09
4	375.81	361.02	361.02	397.79
5	409.91	352.62	365.33	397.79
6	388.69	361.02	369.72	388.09
DRAWN3				
1	379.50	483.72	533.67	518.64
2	391.41	511.36	464.44	511.36
3	406.32	511.36	565.77	490.42
4	420.77	483.72	511.36	549.37
5	404.97	549.37	549.37	533.67
6	409.87	526.08	541.43	511.36

 Table 23.
 Raw data (cont.)

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APPENDIX B

Measurement	Wire type	Kolmogor	ov-Sn	nirnov(a)
		Statistic	Df	Sig.
Diameter(mm.)	COM1	.308	6	.077
	COM1HT	.209	6	.200
	COM2	.173	6	.200
	COM3	.173	6	.200
	GEN1	.313	6	.068
	DRAWN1	.102	6	.200
	DRAWN2	.256	6	.200
	DRAWN3	.134	6	.200
%elongation	COM1	.317	6	.059
	COM1HT	.172	6	.200
	COM2	.158	6	.200
	COM3	.199	6	.200
	GEN1	.196	6	.200
	DRAWN1	.215	6	.200
	DRAWN2	.176	6	.200
	DRAWN3	.155	6	.200
Ultimate tensile strength(MPa)	COM1	.320	6	.054
	COM1HT	.272	6	.188
	COM2	.175	6	.200
	COM3	.205	6	.200
	GEN1	.224	6	.200
	DRAWN1	.191	6	.200
	DRAWN2	.296	6	.108
	DRAWN3	.143	6	.200
Yield strength(MPa)	COM1	.166	6	.200
	COM1HT	.189	6	.200
	COM2	.190	6	.200
	COM3	.224	6	.200
	GEN1	.237	6	.200
	DRAWN1	.188	6	.200
	DRAWN2	.172	6	.200
	DRAWN3	.219	6	.200

Table 24. Test of normality by Kolmogorov-Smirnov.

Measurement	Wire type	Kolmogor	ov-Sm	irnov(a)	
		Statistic	Df	Sig.	
Young'modulus(GPa)-tensile test	COM1	.253	6	.200	
	COM1HT	.150	6	.200	
	COM2	.244	6	.200	
	COM3	.283	6	.145	
	GEN1	.135	6	.200	
	DRAWN1	.210	6	.200	
	DRAWN2	.259	6	.200	
	DRAWN3	.260	6	.200	
Surface Vicker hardness	COM1	.182	6	.200	
	COM1HT	.303	6	.091	
	COM2	.292	6	.121	
	COM3	.183	6	.200	
	GEN1	.264	6	.200	
	DRAWN1	.191	6	.200	
	DRAWN2	.134	6	.200	
	DRAWN3	.244	6	.200	
Cross section Vicker hardness(point 1)	COM1	.240	6	.200	
	COM1HT	.178	6	.200	
	COM2	.289	6	.128	
	COM3	.186	6	.200	
	GEN1	.242	6	.200	
	DRAWN1	.271	6	.192	
	DRAWN2	.299	6	.102	
	DRAWN3	.193	6	.200	
Cross section Vicker hardness(point 2)	COM1	.249	6	.200	
	COM1HT	.226	6	.200	
	COM2	.267	6	.200	
	COM3	.175	6	.200	
	GEN1	.235	6	.200	
	DRAWN1	.306	6	.083	
	DRAWN2	.260	6	.200	
	DRAWN3	.233	6	.200	
Cross section Vicker hardness(point 3)	COM1	.254	6	.200	
•	COM1HT	.222	6	.200	
	COM2	.217	6	.200	
	COM3	.205	6	.200	
	GEN1	.202	6	.200	
	DRAWN1	.232	6	.200	
	DRAWN2	.301	6	.095	
	DRAWN3	.185	6	.200	

Table 24. Test of normality by Kolmogorov-Smirnov (cont.)

		Levene Statistic	df1	df2	Sig.
Diameter(mm.)	Based on Mean	1.331	7	40	.261
%elongation	Based on Mean	19.716	7	40	.000
Ultimate tensile strength(MPa)	Based on Mean	8.371	7	40	.000
Yield strength(MPa)	Based on Mean	2.003	7	40	.079
Young'modulus (GPa)-tensile test	Based on Mean	1.974	7	40	.083
Surface Vicker hardness	Based on Mean	4.206	7	40	.001
Cross section Vicker hardness(point 1)	Based on Mean	1.633	7	40	.154
Cross section Vicker hardness(point 2)	Based on Mean	3.104	7	40	.010
Cross section Vicker hardness(point 3)	Based on Mean	2.115	7	40	.064

 Table 25. Test homogeneity of variances by Levene.

		Sum of		Mean		
		Squares	df	Square	F	Sig.
Young'modulus (GPa)-tensile test	Between Groups	13541.546	7	1934.507	1.910	.093
	Within Groups	40506.697	40	1012.667		
	Total	54048.243	47			
Diameter(mm.)	Between Groups	.001	7	.000	14.148	.000
	Within Groups	.000	40	.000		
	Total	.001	47			
Cross section Vicker hardness(point 1)	Between Groups	453794.879	7	64827.840	342.447	.000
	Within Groups	7572.317	40	189.308		
	Total	461367.196	47			
Cross section Vicker hardness(point 3)	Between Groups	451330.933	7	64475.848	176.691	.000
	Within Groups	14596.318	40	364.908		
	Total	465927.251	47			
Yield strength(MPa)	Between Groups	14405775.728	7	2057967.961	193.131	.000
	Within Groups	426231.863	40	10655.797		
	Total	14832007.591	47			

 Table 26.
 ANOVA for parameter with equal of variances.

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		Statistic(a)	df1	df2	Sig.
%elongation	Welch	10.031	7	16.941	.000
Ultimate tensile strength(MPa)	Welch	10383.852	7	16.157	.000
Cross section Vicker	Welch	374.982	7	17.010	.000
hardness(point 2) Surface Vicker hardness	Welch	20.955	7	16.938	.000

Table 27. Welch test for parameter with no equal of variances.

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BIOGRAPHY

NAME	Mr. Siriwat Chamnunphol
DATE OF BIRTH	26 April 1979
PLACE OF BIRTH	Bangkok, Thailand
INSTITUTIONS ATTENDED	Mahidol University, 1995-2001 :
	Doctor of Dental Surgery (DDS)
	Ramkhamhaeng University, 2001-2003:
	Master of Business administration(MBA)
POSITION & OFFICE	Faculty of Dentistry, Mahidol University
	Bangkok, Thailand