Master Thesis in Biomedical Engineering

Study of the friction coefficient between metallic and ceramic materials for dental implants: fretting phenomenon investigation

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TITLE

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Abstract

Tooth loss is a common issue that can bring the patient to lose the regular aesthetics of the mouth as the correct chewing mechanics. Nowadays dental implants represent a valid replacement of natural teeth.

The most common design for the dental implants involves three different units: the crown usually made in Zirconia, that, thanks to its superior mechanical properties and aesthetics, permit to bear the chewing loads and provides a natural appearance, the abutment, across the gum, usually made in Titanium for is good biocompatibility and the root in direct contact with the bone, also made in Titanium. Despite of that, these two materials have still some problems, mostly related to aesthetic and to the wear caused by their coupling.

In the latest years the dental implants manufacturers focused their attentions more on a form of wear: the fretting. The definition from the ASM Handbook of the phenomenon of Fretting is *"A special wear process that occurs at the contact area between two materials under load and subject to minute relative motion by vibration or some other force."*.[1] This movement condition leads to two forms of damage: superficial wear and fatigue deterioration.

The aim of this study was to evaluate the tribological behavior of three different types of Titanium against Zirconia, to show which of them has the best behaviour under fretting conditions.

Pins made of CPTi, Ti-6Al-4V and DLC (Ti-6Al-4V) were tested against Zirconia balls to reproduce the fixture-abutment contact of a dental implant. The fretting phenomenon was simulated with a Biotribometer (MicroForce, Ducom). To evaluate the different behaviors of the materials, they were tested with 2 different protocols for the Normal load applied: 0.5 and 2 N. Both the tests were run for 40-minutes at a frequency of 1 Hz, with three different steps, in dynamic change, of stroke length: 5 min with 0.02mm, 5 min with 0.05 mm, 30 min with 0.1 mm. The tests were made under the lubrication of BioXtra gel solution, after the ISO 14242-2 2000-2009 cleaning process for the pins with the only use of demi-water.

For every pin was evaluated: the geometric scar wear by means of an optical microscope and the change of roughness with a white light profilometer (profilm 3D Filmetrics). The Coefficient of Friction, during the difference stage of the protocols, was evaluated by means of MATLAB software, as the morphology of the Friction signal.

The data suggest that at a normal load of 2N the DLC (Ti-6Al-4V) is the only one in fretting condition (but near to reciprocating sliding), so the conclusion could be that this material is not a good choice for the dental implant. Despite of that, looking the data for 0.5 N, we can see that the other two materials present an higher profile of fretting.

Putting these observations in relationship with the Coefficient of friction values the DLC (Ti-6Al-4V) appears the only one stable in values and morphology and that makes it the best choice to bear the masticatory cycle (the cycle presents a range of Normal load and different directions of application[2]).

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Chapter 1

Introduction

1.1 History of dental implant

The earliest endeavors of dental implants date back to around 4000[2] years ago in ancient China, where the artifacts found by archaeologists show how the people of that time tried to carve bamboo pegs, tapped into the bone, to replace lost teeth. Around 1000[2] years later in ancient Egypt, similar attempts were made with shaped pegs made of precious metals (Fig 1.1). Proof of this are the mummies found in funerary monuments, which present a large quantity of transplanted human teeth or teeth made of ivory.

Figure 1.1: An Ancient Egyptian dental work with gold wire from 2000 BC

During the 5th century B.C, some writings of Hippocrates, concerning the possibility of anchoring artificial teeth to the gums thanks to the use of metals, were found across Europe. Unfortunately, there isn't a practical confirmation that these writings were also applied.

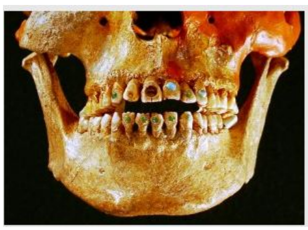

Figure 1.2: A Mayan lower jaw, dating from 600 A.D. with three tooth implants carved from shell

About 1000 years later (around 600 AD) in Central America[3], the first examples of dental implants were found within a population, that, as evidenced by the found artifacts, had great skills in carving and adapting hard stones: the Maya. This population was one of the first to practice dental implants procedures. Using a bow drill, they created perfect holes around the tooth enamel in which they placed carved stones(Fig 1.2). These types of procedures were made more for religious or ritual purposes than for restoring, however they show the Maya were the first to understand how to make dental implant exploiting the osseointegration mechanisms.

The first dental surgical manuals were published only starting from the Middle Age[4], during the 10th and 11th centuries, thanks to the Arabian school, whose major exponent was Abucalsis, one of the greatest surgeons of these ages. A significant part of his work on surgery consists of early descriptions of to dental surgery, describing the procedures for replacing the lost teeth with others naturally or artificially built with bony fragments from large mammals, and the fixing methods with gold ligatures.

Three hundred years[4] later, the french surgeon Guy de Chauliac described an attempt at tooth re plantation in his work *Chirurgia Magna* (Fig 1.3), published in 1363. Afterwards, Michele Savonarola suggested to use ligatures made of linen or silk, for the repair of replanted teeth, and Nicolò Falcucci, illustrated the technique of dental implantation with the aid of metal ligatures.

Figure 1.3: Book cover of Chirurgia Magna of Guy de Chauliac, 1363

During the Renaissance[4], Ambroise Paré, a military surgeon, and The frenchman Dupont were the two leading figures of reference in the field of dental implantation. The first, noted that it was possible to replant teeth, that had been expelled from their sockets accidentally, tying them to the remaining teeth with gold, silver or linen threads, and keeping them tied until stabilization.

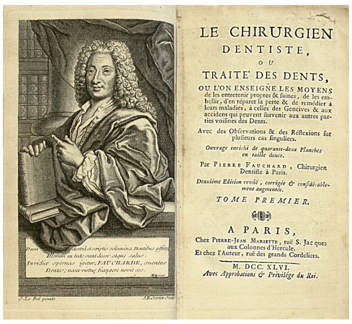

Figure 1.4: Book cover of Le Chirurgien Dentiste, ou Traité des Dents 1728

Dupont introduced an original therapy for pulpitis pain, that consisted in the extraction of the tooth followed by immediate replantation. It is noteworthy that the majority of writings regarding “the right” dental implantation procedure were more theoretical recommendations than the result of clinical evidence because they have not been found data in the literature of that period.

Around the middle of the 18^{th} century[4] Pierre Fauchard, who is considered the founder of modern dentistry, described five replantation cases and one transplant in his work ”Le Chirurgien Dentiste, ou traité des dents” (Fig 1.4). Many others inventors were inspired by the works of Fauchard, for this reason at the end of the 18^{th} century there were many dentistry inventions such as the artificial teeth. Some dentists elaborated different type of couples between different materials to cover the exigencies of good chemical and mechanical properties with aesthetics. It is attested that many different types of artificial teeth were realized using bones/teeth of animals and teeth of the dead, but both materials were of poor quality. Aware of the limits above mentioned, Alexis Duchateau was the first to make porcelain dentures, perfected hereafter by Dubois De Chemant, who adapted them by modifying their chemical composition.

At the beginning of the 1800[5], Rogers, Harris, and Edmons tried intraosseous implants using iron teeth. A decade later Maggiolo, made gold endosseous implants, as reported in his text *Manuel de l’art dentarie*. TThese structures, whose conic root part has three eyelets, were designed to retain the bone; the emerging portion consists of a sort of button, for prosthetic anchorage. In 1891 J.F. Wright designed a porcelain tooth with a porous root area to facilitate engraftment in the alveolus, and later C.E. Friel, provided root holes to Wright's ceramic implant, in order to create drainage in case of development of a periapical abscess. In the first 50 years of the 20th century, there was a large number of attempts for improving the already existent dental implants with the coating or the construction of part in metal of different types. During the thirties, we could observe also the comparison of the Vitalium, a chromium-molybdenum-cobalt alloy, with some positive results, thanks to its great biocompatibility[6]. In 1960 Dr. P. Brånemark (Fig 1.5) was the first to implement the concept of osseointegration, discovered some decades earlier, to the dental practices. This type of approach has brought the dentists to consider also the mechanical properties of the implants for a better fixing and the developments in the design, surgical procedures, and used materials. During the same decade in Japan, Kawahara e Kyocera Co.Ltd. succeeded in the formation of a monocrystalline alumina: this product brought the name of Bioceram and began the first material used for implants in the Nippon country. Beginning in the middle of 1980[7], the used customary implants were the endosseous root-form implant, The factors to consider in order to choose one enodosseous implant over another were: the design, the surface roughness, prosthetic considerations, ease of insertion into the bone, costs and how successfully they were over a period of time.

Figure 1.5: Dr. P. Brånemark 3 maggio 1929 – Göteborg, 20 dicembre 2014

In 1993[7] Dr. David Scharf published in the Journal of Oral and Maxillofacial Implants his dissertation of how the rate of success between implantation in the operating room and in a dental office setting under aseptic conditions is the same. Thus going to change the way of the routine practice in what we have today.

In 2002[7] ADA made a survey about the acceptance of dental implants that showed how it is preferred by the people than tooth replacement. Two years after[7] Genget al.9 made an accurate description of the main category of dental implants with particular attention to the stress distribution and solving problems. The dental implants could be grouped in v-thread,

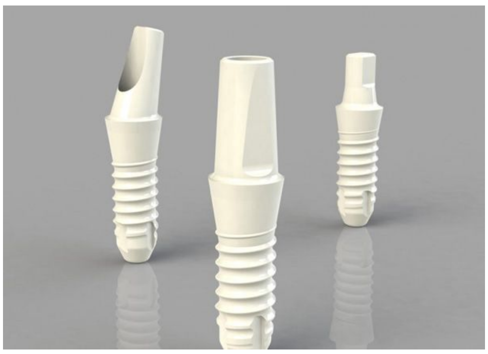

Figure 1.6: Zirconia Dental Implants

thin-thread, reverse buttress, and square thread. New applications in the field were developed by Mehraliet10 in 2013[7] with the development of the design of implants for porous bone with a high biological adaptation(FGMs). In latest years there is also an increase of the use of computer-aided design and computer-aided manufacturing technology for the manufacturing of implants, studying the characteristics of stress distribution in the bone surrounding implants trough computerized three-dimensional models. In the recent clinical studies, Blaschkeet al136 reported that dental implants, made from Zirconia, are a feasible alternative to Titanium dental implants(Fig 1.6). In addition to excellent cosmetic results, Zirconia implants allow a degree of osseointegration and soft tissue response that is superior to that of Titanium dental implants.

1.2 History of Tribology

Over the centuries, the dentists, with the evolving of the technology and the knowledge of the chemical and mechanical principles, have started to develop solutions for the implants with different types of materials. It's clear how in the correct design of this type of devices, also the tribological behavior of the materials used is relevant, because the main purpose is to create an implant with the right biomechanics and that has a long life.

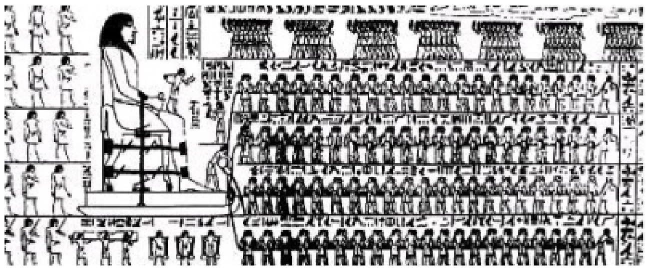

Figure 1.7: Pictorial records about greased skids used to transport an Egyptian statue to the grave of Tehuti-Hetep, El-Bersheh around 1880 B.C

But what is the tribology? *"Tribology is the science of friction, wear, and lubrication and is truly interdisciplinary. It studies the interaction of moving surfaces and every aspect related to friction, wear, lubrication, adhesion, tribochemistry, etc."*[8].

If there is the credence that this study is relatively new, that is a mistak; the first to understand how the different tribological properties of the materials can change the type of interactions of a moving surface were the Sumerians 3000 years ago[9], who for minimizing the friction, thereby reducing the wear and tear as well, lubricated the friction points of the leather loops and of the inverted forks of their carriages.

Around 1000 years after, the Egyptian[9], ancient people of builders, discovered how it was convenient replacing the sliding friction with the rolling friction, increasing the operating speed, solving the problem of friction and simplify strenuous tasks during the transport of huge stone blocks(Fig 1.7). In the first millennium[8] B.C, we can find the earliest documents mentioning the use of wheels to reduce friction and some inventions, especially in the field of the war machines, that take advantage of the principles of tribology.

The Renaissance (15-16th) with the figure of Leonardo Da Vinci, marked the beginning of tribology as a discrete field. He, studying the friction on horizontal and inclined (Fig 1.8) planes, wrote the first two laws of the dry friction.

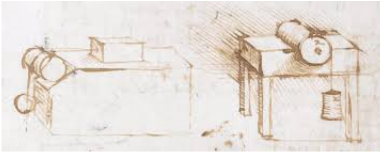

Figure 1.8: Drawings of experiments on friction from Leonardo Da Vinci

The last years of 1600 and the first of 1700 saw numerous scientists trying to improve the understanding of the tribological phenomena, and three of them had a lot of resonance in the scientific community. The first was Guillarm Armonstons[9] who with his studies on the mixed friction found how the roughness of the surface of the materials influences the value of the friction and how the force of this last one depends on the normal force. He elaborated the interlocking theory with two laws, that were only the same two laws that Leonardo Da Vinci previously discovered. The second was Theophilus Desaguliers[9] who developed a tribology model to explain the friction and made some important observations on how the adhesion and cohesion can influence it.

During the same years, Newton[9] defined the material parameter of dynamic viscosity, that gave force to the adhesion theory, making clear how the friction has molecular-mechanical causes.

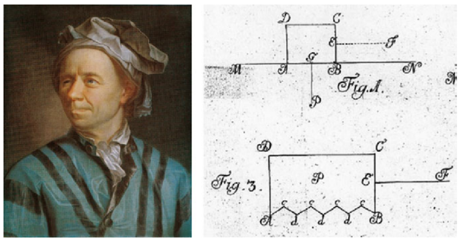

Figure 1.9: : Left-hand side Leonhard Euler. Right-hand side Euler's model of friction

In the 1707 Euler(fig 1.10)[10] made some experiments with tilting inclined planes, discovering that the static friction has to be smaller than the kinetic friction and introducing also a coefficient to describe it "μ". At the end of the 18th century Coulomb[9] continued to develop the basic ideas of Amontons, in terms of surface roughness and mixed friction. According to Coulomb's model, the load applied on the surface area does not influence the friction coefficient of a given surface and the friction force is proportional to the weight of the object that is sliding. According to that, the sliding friction is not an energy-consuming process, incorrect assumption then proved. With the Industrial Revolution[9], there was also an increment of demand for lubricants in terms of quantity and quality. These new needs to supply and the discoveries in fluid mechanics and viscous flow properties brought to advances in the development of lubricants and to a rapid replacement of vegetable and animal oils with mineral oils. In the 1798[11], there was the first reliable test on frictional wear, conducted by Charles Hatchett and Henry Cavendish, using a reciprocating machine to evaluate the wear rate of gold coins.

Then another period of "technical progress" started from 1850 going to increase in the 20th century, an increasing request for automation process brought also to a big demand of lubricants to avoid the wear of the industrial machinery elements and improve the efficiency. Thanks to these necessities there was, in the field of the lubricants creations, the development of new synthetic synthesis and the adoption of additives for improving the lubricants properties[12]. During these years the relationship between friction, wear and lubrication were investigated by many scientists, especially in the applied science to journal bearings. The most famous were: Heinrich Rudolf Hertz with his work on the physical laws of rolling friction and Tower and Reynolds with their studies on the hydrodynamic pressure related to the bearings[13].

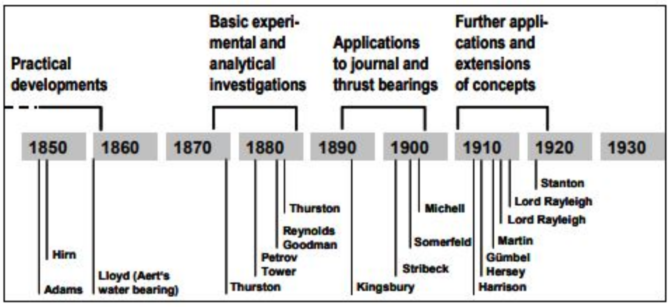

Figure 38: Main scientists involved in theoretical aspects of bearings

Figure 1.10: : Main scientists involved in theoretical aspects of bearings

After them, many scientists studied how to improve or extent the preexisting theories by others(Fig 1.10)[13]. In the 1950[13] Bowden and Tabor wrote "The Friction and Lubrication of Solids" a book that covers the behavior of non-metals, especially elastomers, elastohydrodynamic lubrication, and the wear of sliding surfaces. The theories elaborated by them gradually replaced the earlier concept of the friction mechanism. Theirs contribute to the adhesion theory in the field of tribology is one of the fundamental parts of the friction theory. Immediately, after few years, Archard developed the wear law for the sliding contact and write large quantities of articles and books based on tribological behavior of "Rubbing Surfaces". In 1966[10] the term tribology was mentioned for the first time in a study, commissioned by the British government to investigate the damage from wear. In the report it was described the connection between friction, wear and lubrication in the damage phenomena[12]. Since the latest decades of the 20th century[14] till now the globalization and the increasingly competitive markets have pushed the researchers to develop synthetic based oils with high performance for a use in condition of high / low temperature and high load. This trend is explained by the need to improve the life or life service abilities of the machines in the factories, and by the need to consolidate and improve the production techniques of synthetic base oils already existing. Some highlights shall be mentioned about this period: the approximate solutions of the Reynolds equation (e. g. Michel OCVIRK, Du BOIS, KINGSBURY, CAMERON, SASSENFELD/WALTHER) and their application to the hydrodynamic operating machine elements, the elastohydrodynamic solution of the Reynolds equation (e. g. Duncan DOWSON together with HIGGINSON), and the Application of EHD to the calculation of highly loaded lubricated contacts.

1.3 Tribology concepts

In the history the achieved discoveries could be grouped in three main branches: friction, wear, and lubrication.

1.3.1 friction

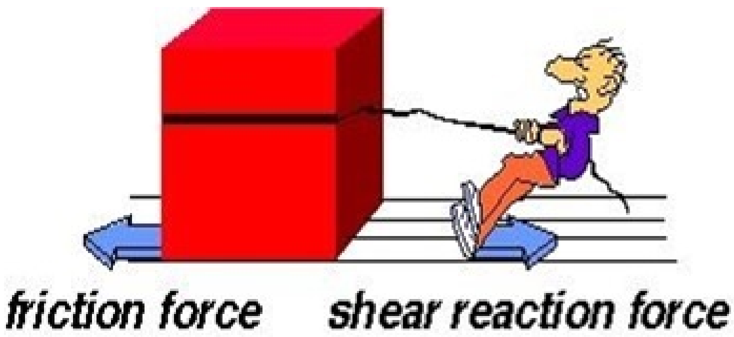

Figure 1.11: Representation of Friction force

Friction *"is a force acting opposite to the direction of relative motion"*[15](Fig 1.11). This type of force is the resultant of electro-magnetic and gravity forces and its nature could be of different type (deformation of the surfaces, adhesion, etc). In every energy process where there is a friction force, there is always energy dissipation and the modern tribology studies and exploits this characteristic, for developing new way to decrease the wear in the machines. Talking about the dry friction there are three laws of friction[15]:

1. Amontons' First Law: The force of friction is directly proportional to the applied load[15].
2. Amontons' Second Law: The force of friction is independent of the apparent area of contact[15].
3. Coulomb's Law of Friction: Kinetic friction is independent of the sliding velocity[15].

This 3 different laws could be good resumed by the physics formula:

$$F = \mu * N \tag{1.1}$$

Where N is the Normal load applied between the surfaces of the materials and μ a dimensionless factor, called coefficient of friction. This coefficient could be either static, if there is no relative motion, or kinetic, if the motion is happening, typically the first is always bigger than the second. Usually, for distinguishing the different tribological properties of materials,μ is calculated to be used as a term of comparison.

1.3.2 wear

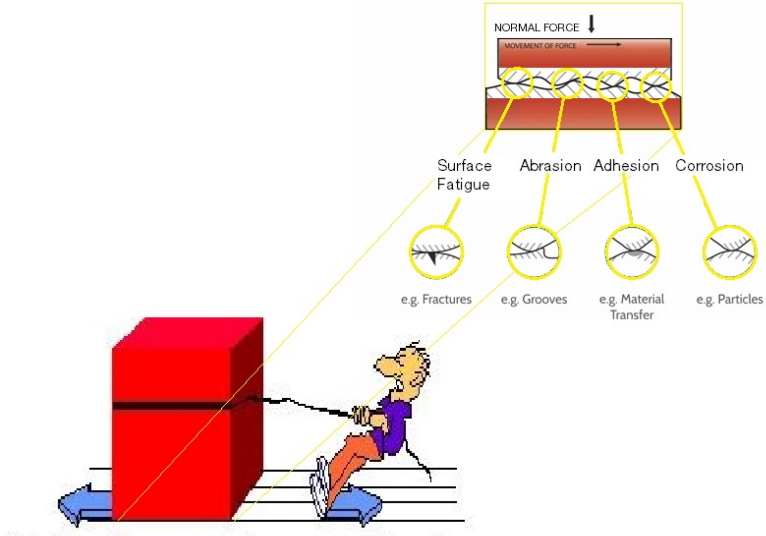

Figure 1.12: Representation of the different types of wear

The wear is defined as a progressive loss of substance from the surface of a body of one or all solid surfaces in contact subjected to relative motion. This substance is the result of one or more different patterns corresponding to various wear mechanisms, which can change with the flow of the time or adjustement in operational conditions. These mechanisms can be distinguished in four different groups (Fig 1.12)[16]:

1. Adhesive: this wear is the result, in term of loss of substance, of two surfaces under mutual sliding or that are pressed one against the other(in this type the roughness of the surfaces play a main role).

2. Abrasive: it occurs whenever a soft material is plowed by a rough hard surface (a lot of surface finishing techniques take advantage of this phenomenon).

3. Surface fatigue: the wear is related to the number of cycles that the materials have to undergo.

4. Corrosive: this is a process very quick and is related to chemical reaction with the environment. This type of chemical-mechanical degradation is usually very quick at the beginning and tends to slow down afterward because the phenomenon tends to reach a chemical balance.

This division is a rough classification because the wear is influenced by many factors that are related by the type of motion, the type or mode of wear and its basic mechanisms of it. Ko[17] in his work has grouped the relationships between them in the following scheme (Fig 1.13).

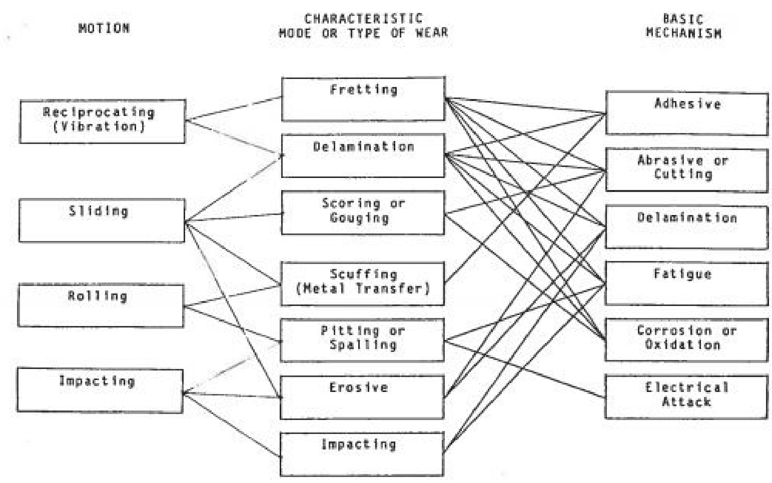

Figure 1.13: the relationship between motion, mode, and type of wear, and basic mechanism of wear by Ko

1.3.3 lubrification

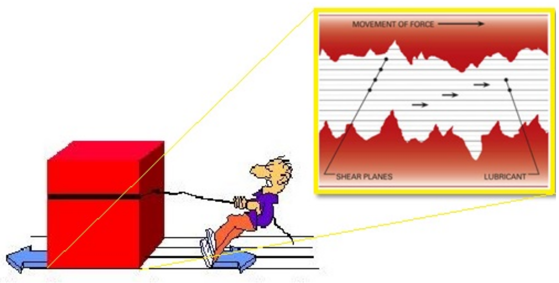

Figure 1.14: Rappresentation of Lubrication

A lubricant (Fig 1.14) is a substance used to reduce friction and wear, used for increasing the lifespan of the operating elements, which have surfaced in a condition of sliding contacts[18]. To do that, the tribologycians use the equations of the theory of hydrodynamic lubrication or of the theory of elastohydrodynamic lubrication. The lubricant can be liquid, gaseous, solid or a combination of them, in form of emulsion or grease[19]. Four different forms of lubrication can be identified by self-pressure generating lubricated contacts[19]:

- Boundary: is characterized by close contact between the asperities. In this condition, the physical and chemical properties of the surface in contact are important, because the load is entirely supported by the asperities while the lubricant function is practically useless.

- Hydrodynamics: it is a stable condition in which the lubricating layer fully supports the applied load through the development of a pressure. This last one is generated by a movement at a sufficiently high speed of the surfaces, which allows to separate them.

- Elastohydrodynamic[19]: in this regime the surfaces tend to deform during the hydrodynamic action due the high load over them.

- partial or mixed: in this regime, the tallest asperities of the bounding surfaces protrude through the film and occasionally come in contact between each other, that is related to a situation where the speed of the movement is low, the load applied is high or the temperature is sufficiently large to significantly reduce lubricant viscosity

The Stribeck-curve below (Fig 1.15) shows how these lubrication regimes are related to the Friction.

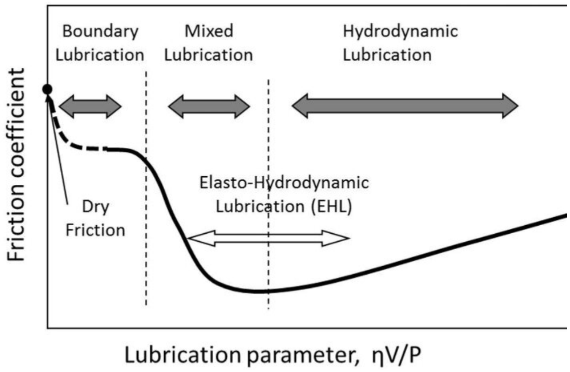

Figure 1.15: Representation of The Stribeck-curve of the different lubrication regimes

1.4 Biotribology

1.4.1 introduction to biotribology

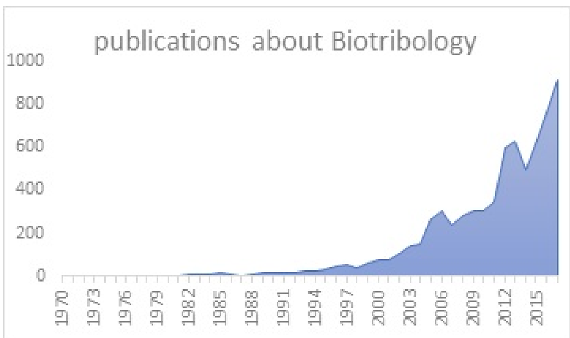

Figure 1.16: Number of Publication of Biotribology from 1970 to now by google scholar

During the history, the tribology has seen its development related to the major mechanical inventions in the industries. But also in the field of bio-system there were many investigations concerning the lubrification, the wear, and the friction. Despite of that, the first time that the word Biotribology was used was by Dowson in 1970[14].

Since that moment the interest in this field, as it's showed in the graph extracted with google scholar (Fig 1.16), the biotribology investigations have undergone an exponential growth in the number of publications trhough out the years, that is strongly related to new technology developments for the health caring and the device/technologies human based.

Z.R.Zhou[13] offers in one of his work a classification of the current investigations on biotribology according to different subjects or systems from various conferences or journals. The table extracted (Fig 1.17) by his work shows that in the oral tribology the implant teeth is one of the main fields of research.

Classification type	Major investigations
Joint tribology	Hip joint; knee joint; articular cartilage; joint fluid; restorative materials of joints; implant interfaces; etc.
Skin tribology	Skin friction-induced perception; skin care; synthetic skin; skin in contact with articles (such as tactile texture, shaving devices, shoes, socks) for daily use, various medical as well as sports devices, medical and cosmetic treatment; skin friction and grip of objects; skin irritation and discomfort; etc.
Oral tribology	Natural teeth; tongue; mandibular joints; saliva; implant teeth; toothpaste; swallow; dental restorative materials; etc.
Tribology of the other human bodies or tissues	Hairs; bone; cells; contact lenses; ocular surfaces; capillary blood flow; etc.
Medical devices	Scalpel; operation forceps; urinary catheters; gastroscope, artificial cardiovascular system; medical gloves; etc.
Animal tribology	Gecko adhesion; animal locomotion; pangolin scale; the skin of fish and shark; feather of birds; water strider; earthworm; ants; beetle, butterflys wing; seashell; snails; etc.
Plant tribology	Lotus leaf; diatoms; etc.

Figure 1.17: Table of a major investigation in the field of tribology by Z.R.Zhou

1.4.2 dental tribology

With the aging, the teeth are going into a natural retention process, and consequently, in developing different forms of wear[20]. All of them are related to the habits and to the clinical history of the patient. The normal development of wear is the Physiologic wear, that is a natural consequence of the vital functions of human mouth like the mastication, that results in the slow reduction of the convexity of tooth cusps. If this wear is excessive and caused by some alteration of the oral physiology, is called Pathological wear. In addition to these, there are also other two types of wear that are related to the human intervention: the Prophylactic wear for a preventive purpose of maintaining a good hygiene of the mouth, like the toothbrushing, and the Finishing procedure wear, that is the result of different processes during the dental restoration.

Wear of human teeth and dental implant

The development of wear of teeth is related mainly to the mastication. It has an open phase, in which there are no occlusal forces, and a closed phase, where an occlusal load is applied to the occlusal surfaces of opposing teeth or to the food between them and the particles between the opposing surfaces. There are three main mechanisms that produce the loss of dental hard tissues of the teeth[21]:

- erosion, that is produced by the chemical dissolution of enamel or dentin caused by the action of non bacterial acid from dietary or gastric sources.
- attrition that is the physiological wear due to tooth-to-tooth contact.
- abrasion that is a pathological wear caused by abnormal mechanical processes that involve foreign objects or substances that are repeatedly put in contact with the teeth.

Looking at dental implants, during mastication they are exposed to mechanical actions, causing micromotion (fretting movements), temperature fluctuations, oxygen pressure variations, and a chemical environment. All these phenomena conduct to a continuous complex degradation process[22].

Lubricating effect of saliva

During mastication, tooth wear generally occurs with the lubrication of saliva or food slurry[20]. Saliva is an exocrine solution mainly composed by water (99%), the remaining 1% consists of a wide variety of electrolytes and proteins, which determine its functions and properties. Its main function is to form a boundary lubrication system and to serve as a lubricant between hard (enamel) and soft (mucosal) tissues.

The saliva helps to decrease the wear of teeth, thanks to its reduction of the coefficient of friction of the oral mucosa and of the tongue surfaces. Besides that, its lubricating characteristic allows to prevent lesions, to swallow easier, and to speak[20]. The lubricating effect of saliva is however influenced by the properties of the substrate surface, as the roughness, the free energy, the chemical composition, and the electrical charge[20]. Therefore this aspect is taken into consideration by the clinicians during the design phase of the implants.

Chapter 2

Dental implants

2.1 Anatomy of a tooth

Before to talk about dental implants a quick review of the teeth anatomy could be interesting for a better understanding of future concepts. The teeth are very important for our health, but they also play an important role in phonation and social life. The teeth that make up a dentition always depends on the person but generally in an adult the number is quite the same: 32. They are fundamental for chewing food and their functions are closely related to their form and structure. We can thus identify four dental groups(Fig 2.1):

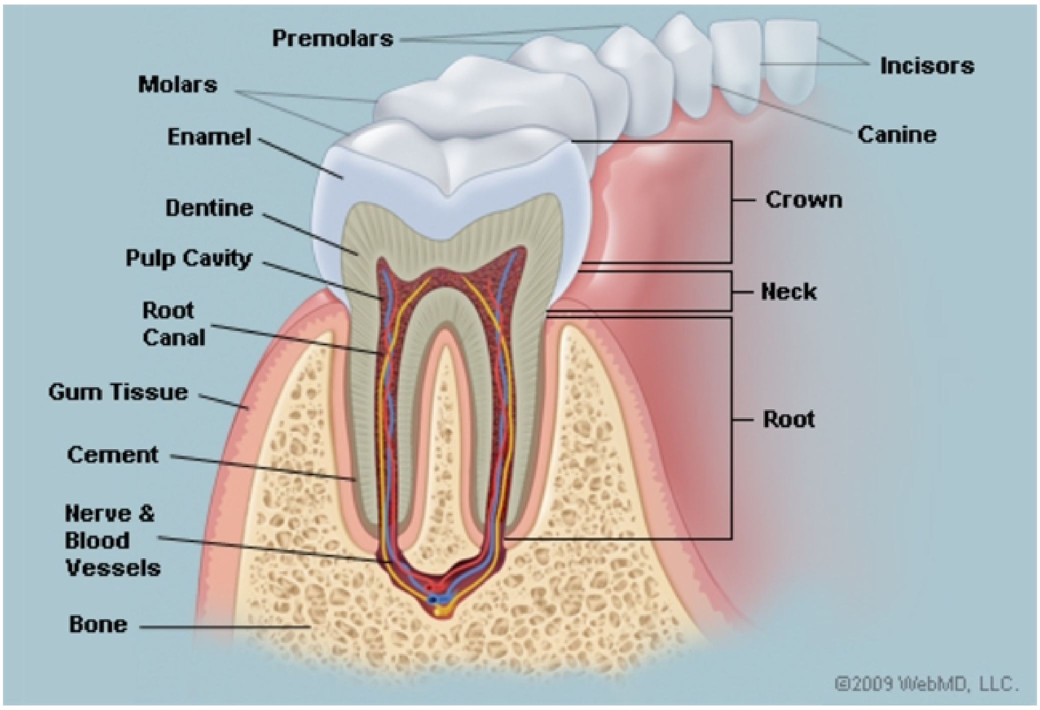

Figure 2.1: Structure of a tooth and different types of it

1. Incisors (8 teeth): these are the teeth in the front and middle of the mouth, four in the upper part and the other four in the lower part. They are the ones we use to cut food.

2. Canines (4 teeth): these are the sharpest teeth and serve to tear the food.

3. Premolar (8 teeth): also called bicuspids, they are used in the first phase of mastication, for the first grinding of food.

4. Molar (8 teeth): used to grind food.

In addition to these 4 groups, there is a fifth one that not everybody has and about which we have already spoken in our previous post. These are the tertiary molars (4 teeth) commonly known as wisdom teeth.

2.1.1 Structure and functions

Regardless of its shape and function, each tooth can be divided into three parts(Fig 2.1):

1. ROOT: it is the part located inside the alveolar bone, that is the part of the tooth that is not seen. It's made up of several parts:
   - Root canal: it is a passageway that contains pulp.
   - Cementum: it is bone-like material that covers the tooth's root.
   - Periodontal ligament: it is a connection between the teeth and the tooth sockets made of connective tissue and collagen fiber, that contains both nerves and blood vessels.
   - Nerves and blood vessels: the first help to control the amount of force during the chewing, the seconds allow to supply nutrients to the periodontal ligament.
   - Jaw bone: it is the bone that holds the teeth in place, contains the tooth sockets and surrounds the teeth's roots.

2. NECK: it is the intermediate part of the connection between root and crown, around which the gingiva margin develops. It has three main parts:
   - Gingiva: it is a soft tissue that surrounds the teeth and covers the alveolar process..
   - Pulp: it is the innermost portion of the tooth. It's made of tiny blood vessels and nerve tissue.
   - Pulp cavity: The pulp cavity, sometimes called the pulp chamber, is the space inside the crown that contains the pulp.

3. CROWN: The crown of a tooth is the portion of the tooth that's visible. It contains three parts:
   - Anatomical crown: this is the top portion of a tooth, the only part of a tooth that we can see.
   - Enamel: is the substance that covers the area of the tooth exposed to the oral environment. It is not a tissue, but a tissue derivative produced during amelogenesis by cells of epithelial origin.
   - Dentin: is a particular form of bone tissue, harder than compact bone, which is part of the compact substance of the tooth that surrounds the pulp cavity.

2.2 Introduction to dental implant

With implantology, we define that set of surgical techniques designed to functionally rehabilitate a patient affected by total or partial edentulism, through the use of dental implants, surgically inserted into the mandibular or maxillary bone. These implants can be of different shapes, inserted in different sites with different techniques and then connected to the prosthesis with different timing.

2.2.1 Types of dental implants

The systems can be divided into three types[23]:

1. Subperiosteal implants (Fig 2.2): a metallic structure is placed surgically between the surface bone and the periosteum, then immediately above the bone; the pillars of the implant transpire in some points of the mucosa that covers it. These implants are rarely used, because their shape is not rather similar form to the natural roots, therefore less reproducing the right stimulus to osseointegration that results in high percentages of failure.

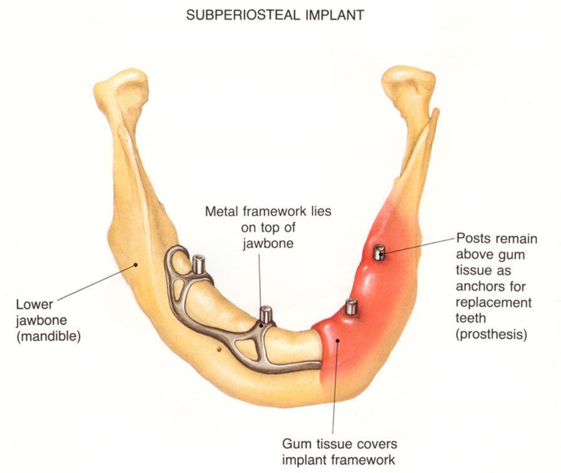

Figure 2.2: subperiostal implant

2. Endosseus implants[24]:

   - Blade(Fig 2.3):were developed in the 1960s. Although they have been

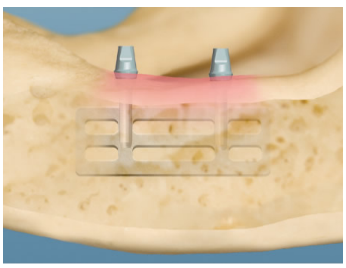

Figure 2.3: blade endosseus implant

successful for many years, they need for high surgical skills and the lack of osseointegration, high morbidity and frequent postoperative complications have led to the interruption of its use.

- Root form[25]: a cylindrical core is inserted directly into the cavity of a fresh extraction, or into a hole obtained directly from the bone for drilling. They could be divided in two different types that are different by the procedure of installation:
  - Single stage(Fig 2.4)[25]: The implant, which is left transmucous, emerges. It can be allowed to heal (2-6 months) for bone integration or loaded immediately (one-stage implants with immediate loading).

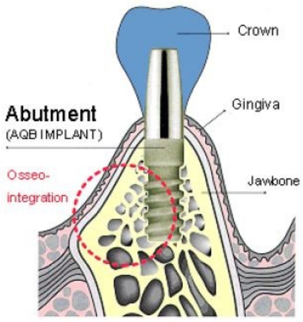

Figure 2.4: one stage endosseus implant

  - Two stages(Fig 2.5)[25]: the first "submerges" the implant insertion with a submucosal suture, the second consists in reopening the mucosa after 2-6 months and screwing of the "dental pillar" on the implant.

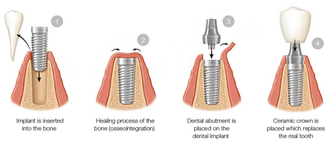

Figure 2.5: two stages endosseus implant procedure

3. Transosteal implants(Fig 2.6):The implant crosses the entire thickness of the bone until a segment comes out and allows fixing with a plate. This type of implant is used when the seal cannot be guaranteed by a simple osseointegrated implant.

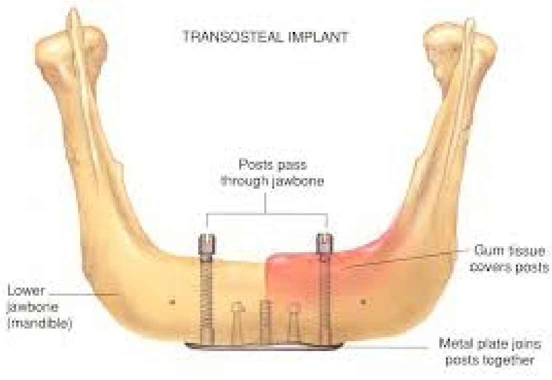

Figure 2.6: transosteal implant

2.2.2 Implant components

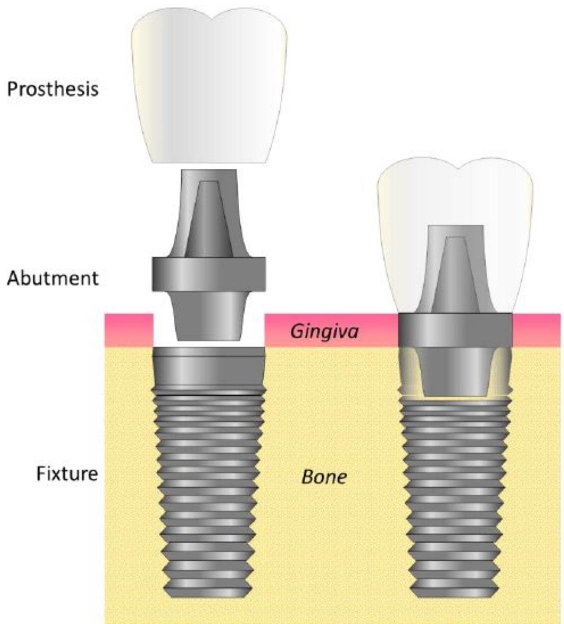

Figure 2.7: components of endosseus implant

Despite the large variety of designs, the main components of a dental implant are essentially unchanging and they are(Fig 2.7): a body, a transmucosal abutment, and a prosthesis. The body is essentially an endosseous screw, called fixture, and it is the element of the cylindrical and threaded dental implant, which, following its insertion into the mandibular or maxillary bone, will undergo the osseointegration process. The abutment is the element of the dental implant that connects the endosseous screw to the dental prosthesis. After insertion of the dental implant, it represents the portion in close contact with the gingival mucosa; this explains why it also takes the alternative name of the transmucosal component. The dental prosthesis is what replaces the missing or extracted teeth. In fact, it is the external part of the dental implant, which has the task of covering the toothless area restoring the biomechanical and aesthetic functions.

2.2.3 Dental implant materials

Looking at Material that can be used for a long time in contact with body tissues without causing rejection reactions. In the dental field, they can be divided into three categories of biomaterials according to their application in dentistry[24]: *restorative*, used for replace a missing or a malfunctioning tooth structure, *auxiliary*, that facilitate or are fundamental for the fabrication of prostheses, and *preventive* materials, that inhibiting the tooth degeneration or even preventing it. During the course of history, as we have seen in the previous chapter, the people used various materials in the past to restore or repair teeth. nowadays the dental materials used in the implant are almost metals, ceramics, polymers, and composites. Here below, according to Reham B. Osm and Michael V. Swain[26] in their work, there is a table of the principals materials used for the components of a endosseus implant(Fig 2.8).

Implant Material	Common Name or Abbreviation
I. Metals	
Titanium	CpTi
Titanium Alloys	Ti-6A1-4V extra low interstitial (ELI)
	Ti-6A1-4V
	Ti-6Al-7Nb
	Ti-5Al-2.5Fe
	Ti-15 Zr-4Nb-2Ta-0.2Pd
	Ti-29Nb-13Ta-4.6Zr
	Roxolid (83%–87%Ti-13%–17%Zr)
Stainless Steel	SS, 316 LSS
Cobalt Chromium Alloy	Vitallium, Co-Cr-Mo
Gold Alloys	Au Alloys
Tantalum	Ta
II. Ceramics	
Alumina	Al2O3, polycrystalline alumina or single-crystal sapphire
Hydroxyapatite	HA, Ca10(PO4)10, (OH)2
Beta-Tricalcium phosphate	β-TCP, Ca3(PO4)2
Carbon	C
	vitreous,
	low-temperature isotropic (LTI),
	ultra-low-temperature isotropic (ULTI)
Carbon-Silicon	C-Si
Bioglass	SiO2/CaO/Na2O/P2O5
Zirconia	ZrO2
Zirconia-toughened alumina	ZTA
III. Polymers	
Polymethylmethacrylate	PMMA
Polytetrafluoroethylene	PTFE
Polyethylene	PE
Polysulfone	PSF
Polyurethane	PU

Figure 2.8: Table of materials used for endosseus implants according to Reham B. Osm and Michael V. Swain

Metallic biomaterials

In the design of the dental implants, the metals are largely used, or as main elements for the structure of one or more components or as a coating of them[25]. The chosen material used for dental implants is titanium in its commercially pure form (CPTi4), as a material with optimal characteristics such as mechanical resistance, low density, relatively high flexural strength, excellent corrosion resistance combined to high biocompatibility, or more properly bio-inertia[27]. Stainless steel[25] (approximately 20 % chromium and less than 10% nickel) in its surgical austenitic form, has a very low rate of use, because, despite its low cost and easy fabrication, it presents some disadvantages that do not make it perfectly suitable, as its prone to elicit allergic reactions, its susceptivity of crevice and pitting corrosion. The Cobalt-chromium-molybdenum alloys, like Vitalium, have good mechanical properties and, thanks to the Chromium, high resistance to corrosion. In the 1930s[25] it was introduced as new biomaterial for dental implant because of its biocompatibility and the electrochemical stability, but after some years it was proven responsible of chronic inflammation and lacking in osseous integration.

Ceramic Biomaterials

Ceramic materials can exist in a wide variety of forms, both crystalline that glassy (or amorphous). The atomic structure of ceramic materials (nature of the chemical bond and micro-structure) gives them the properties of excellent resistance to attack of chemical agents, electrical and thermal isolation, and good resistance to compression loads. Unfortunately, they have a mechanical behavior characterized by fragility: the crystalline structure of ceramic materials not allows the relative movement of atoms, which means that when the material is deformed beyond a certain limit, it suffers a fragile fracture. Alumina (aluminum oxide, Al_2O_3) is used in dental application in the form of single alumina crystal. This material has an high biocompatibility and an high bioinertia, thanks to that, it remains stable for many years after implantation [28]. Zirconia (zirconium oxide, ZrO_2)[28] is a very suitable material for construction of dental prostheses, thanks to its particular characteristics. It is used in the form of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP)[28] that is extremely biocompatible, has high flexural strength as well as superior corrosion and wear resistance. Another very important feature of this material is the translucency, which makes its appearance very close to the natural teeth.

2.3 Requirements for Successful Implant Systems

According to Yoshiki Oshida, Elif B. Tuna, Oya Aktören and Koray Gençay[27] the three main aspects to follow during a design and validation of a "successful implant system" are:

- SAFETY: all the implants must be designed following the guidelines of the medical device amendment of FDA27 and be classified according to their grade risk for the health of the patient. Their intrinsically safety and effectiveness must be checked by the different test suggested in the ISO standards[27].

- COMPATIBILITY: being the implant created for the purpose to reestablish the correct function of the oral component damaged, it must be designed with appropriate mechanical and morphological properties and be biocompatible*(Fig 2.9)[29].

  Knowing that the materials will undergo in contact with living tissue and so will influence it, the first thing to check is the biocompatibility[27]. Nowadays

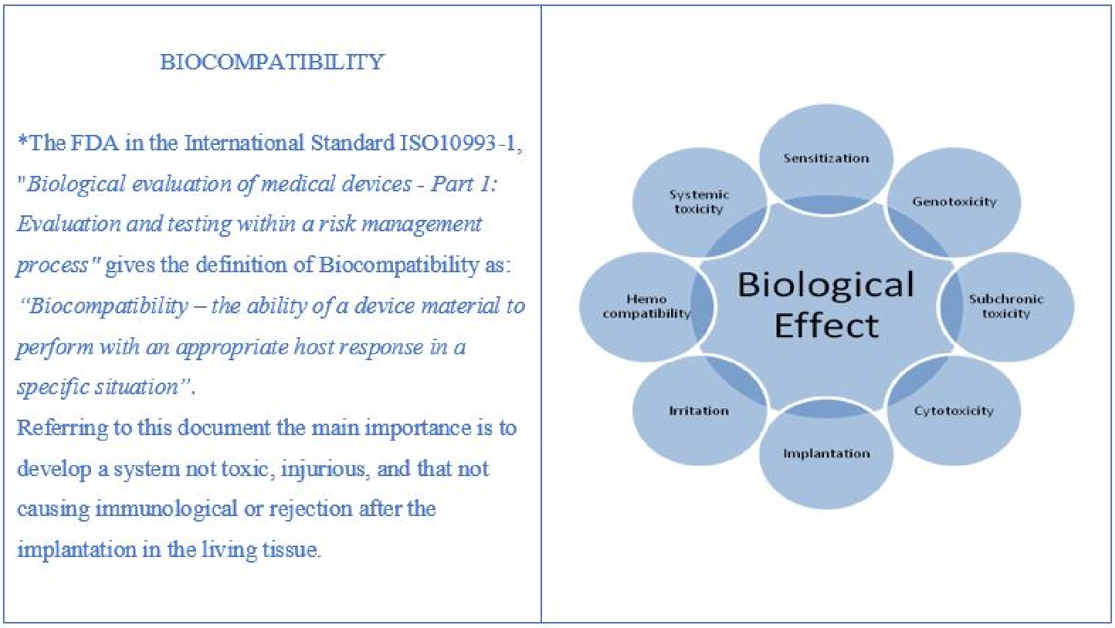

Figure 2.9: biocompatibility aspects

the main materials used for building implants release ions (especially metals) of different nature that could cause inflammatory process, cytotoxicity and their discoloration, so it's good practice to choose materials extremely biocompatibile and with a high value of resistance to corrosion[27]. As concern the mechanical part the implant must be designed to be efficient and with long service life. To do that the clinician has to study the types of the biting forces applied on the implant, how them are transferred to the interfacial tissues, and how these least react to them [28]. The morphology results also important for a successful implant, because the macro and microstructure influence the osseointegration*(Fig 2.10) process, the chemical surface interaction, and the biomechanical sollecitation[27].

A good surface morphology usually has an upper and lower limitations in average roughness (0.001-0.05 mm) and average particle size. These aspects as the biological reactions can be studied in three different zone of interactions[29]:

1. *Bone / fixture interface.*
2. *Abutment / fixture / Soft Tissue Interface.*
3. *Abutment / Prosthesis interface.*

1. *Bone / fixture interface*

   The healing process of the tissues around the dental implants involves the osseointegration of most of the implant surface leaving a small percentage available for the remodeling. Subsequently, the application of the chewing loads stimulates the remodeling and maturation of the bone tissue. At the time level there are three different phases[29]: (0.010-0.500 mm) [27]. The term osseointegration, coined by Per-Ingvar Brånemark, is used in dentistry and medicine to define[30] the intimate union between a bone and an artificial implant without apparent connective tissue. Intimate

OSSEOINTEGRATION

The term osseointegration, coined by Per-Ingvar Brånemark, is used in dentistry and medicine to define[30] the intimate union between a bone and an artificial implant without apparent connective tissue. Intimate union is defined when space and relative movements between bone and implant do not exceed 100 microns[30]. This phenomenon depends closely by the adequate micromorphology and the absence of proteins of the material. According to that the speed of the osseointegration process and its quantity vary according to the type of implant surface[30]. Smooth surfaces are less suitable and nowadays for this reason are used to improve the osteointegration special treatments of surface finishing or surface coating[30].

Figure 2.10: osseointegration of a dental implant

union is defined when space and relative movements between bone and implant do not exceed 100 microns[30]. This phenomenon depends closely on the adequate micromorphology and the absence of proteins of the material. According to that, the speed of the osseointegration process and its quantity vary according to the type of implant surface[30]. Smooth surfaces are less suitable and nowadays for this reason they are used to improve the osteointegration special treatments of surface finishing or surface coating[30].

- primary stability: it is obtained during the first surgical phase and is necessary to avoid micromovements, and so the fibro-integration, forming a bone matrix[29].
- secondary stability: the new bone matrix, created in the previous phase, undergoes progressive mineralization and maturation[29].
- maintenance of osseointegration under functional load: the transmission of masticatory loads to a osseointegrated implants is characterized by significant biomechanical differences in confront to natural teeth[29]. For example, the occlusive force exerted on the implant is more damaging than the natural tooth, because it is transmitted directly to the bone tissue without the distribution by the roots and without the feedback mechanism of the mechanical receptors[29]. The fixture must therefore be designed to achieve stability in such conditions, not exceeding the breaking loads and fatigue limits[29].

2. *Abutment / fixture / Soft Tissue Interface* The abutment-fixture connection is a fundamental part of the distribution of masticatory loads from the prosthesis to the bone-fixture interface as it represents a point of discontinuity and weakness of the system[29]. Ideally, a connection should be precise, to ensure the maximum possible seal between abutment and fixture, going to prevent a possible bacterial formation, and

stable, to ensure adequate resistance to masticatory stress, without the two connected components having movements relative one over to the other, possible source of usury[29]. The connection must therefore resists the phenomenon of static static type breakage, to achieve a local stress value equal to that of the elastic limit of the material, or dynamic break, resulting from repeated application of the load, even for relatively modest loads, leading to the formation of cracks[29]. This happens generally when the repetition of the load cycles does not take place with fixed periodicity, nor between precise values but in a casual way: the effect is always the same. It is verified that, due to possible defects that are always present inside the material or on its surface, the distribution of the stress can grow[29]. Therefore the design and construction must take into account not only the maximum forces that can be applied, but also the many applications to which the product will meet in its life[29].

3. *Abutment / Prosthesis interface.* In the zone between prosthesis and abutment is possible to find a multiple-fixture force transmission, due to micro-movement caused by the deformation of the retaining screws, and possible overload, caused by poor interface fit between prosthesis and abutments that could result in the loosing/ damage of the implant[31]. Since the transmission and distribution of these are important for a correct integration and durability of the implant, the prosthesis must follow two basic requirements: precision of fit, to ensure a correct marginal closure on the abutment to limit the marginal bacterial infiltration and passivity: it does not exert traction on the fixture and therefore on the bone tissue[**29.7**]

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- MRI SAFETY AND IMAGE COMPATIBILITY: being the change of magnetic field to the base of technology of the MRI, the clinicians during the design of the implant must to pay attention to the geometry of it[27]. That because both magnetic and non-magnetic metallic devices may be subjected to heating or cause image artifacts due by the interactions with the magnetic field, compromising so the procedure and image quality[27].

Chapter 3

Experiment

3.1 identification of a need

Nowadays titanium dental implants are the most common solution for the replacing teeth holding around the 97% of the market[32]. This type of implant is subject to two principal problem[32, 33]: the hypersensitivity, caused predominantly by a patient's titanium allergy and that induces a consequent implant failure inhibiting the integration, and the greyish appearance of the tissue, that is the combination of the release of titanium ions and a thin patient's soft tissue, which result in poor aesthetic results[33].

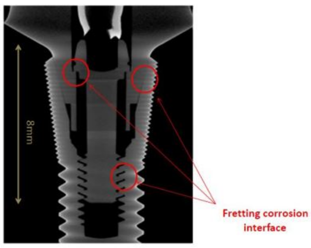


Figure 3.1: fretting corrosion in the fixture/abutment interface

The 3Y-TZP is a ceramic material typically used for crowns, and now, to solve the problem cited before, it is used also for the abutments, improving so the appearance and hipersensibility problems respect to the all-titanium implants[32]. Beside of that, the hardness of this material is higher respect the titanium one and consequently the coupling of them making this last to incur to wear in case of mutual micronmovements[33].

Micron mobility is when the amplitude of the movement is less than 100 μm. In dental implant this movement coincides with a sliding called fretting[34]. Fretting is a type of damage, which occurs between two surfaces placed in contact with each others, under the action of a normal force and subjected to cyclic low-amplitude oscillatory sliding[35, 36].

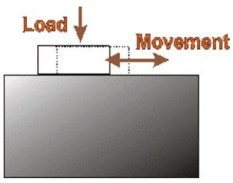


Figure 3.2: fretting phenomenon

When the number of cycles is big enough, the material develop cracks over the surface and result in fretting fatigue, and in case of the dental implants it results in rupture or prosthetic detachment or in need of re-operation. Fretting can greatly reduce the fatigue life of the contacting parts(Fig 3.1)[34, 37].

Nowadays the major interests are related to supply the problems described above, which usually converge to develop new type of couple between materials, coating treatment and different type of geometry of the implant pieces[29, 32, 33].

3.2 definition of the problem

3.2.1 The Fretting

According to the work of Vingsbo and Soderberg there is no a limit value of the "small amplitude", but in general the literature suggests that the upper transition amplitude is around the 150 - 300 μm[38](Fig 3.2). For what concern the lower limit, where the fretting damage doesn't occur, in literature there are no fixed values[38]. It is therefore difficult to resolve the definition of limit parameters of the phenomenon because they are the result of a mix of different operating parameters, but the type of surface damage are easily divided in three different categories[38]: fretting fatigue, fretting wear and fretting corrosion.

1. Fretting fatigue: It is a damage phenomenon of fatigue on the surfaces of the materials due to cyclic stress in fretting condition[39, 40]. The crack growth behavior of this damage is different in comparison to the friction-less fatigue. That because its propagation, near the contact surface, decreases with the increasing of its length[39]. Despite of that, the fretting fatigue strength is often half or less of the fatigue strength friction-less and it's not influenced, even if the tensile strength of the material increase[41]. It follows that the fatigue life is less in a fretting situation and even if the stress amplitude is under the value of the fatigue failure. Consequently the crack will nucleate, due to friction, and will grow resulting to the failure of the implant[42].

2. fretting wear: It is a damage phenomenon in the form of a combination of abrasive and adhesive wear, that occurs between two surfaces placed under fretting condition[39, 42]. It causes rupture of the material in a form of debris, that will remain entrapped in the contact zone, forming a sort of bearings, that can protect the material from the wear[43]. This may lead to the reduction of the coefficient of friction with the flowing of the time.

3. fretting corrosion: It is a damage phenomenon that occurs between two surfaces placed under fretting condition and when between them there is the presence of a corrosive medium[44]. For this type of fretting damage can be distinguished three different phase[41]: initially with the breaking of the oxide layer the two surface come in contact in a partial / mix lubricated condition (cold welding, adhesion)[18, 44]. Then the contact between the asperities creates debris that remaining entrapped in the zone and consequently oxidized, this last will act as abrasive increasing the wear rate. Finally it converges in a fretting fatigue phenomenon[45].

The stick-slip phenomenon

At the base of the fretting damages there is the stick slip-phenomenon(a engineering representation in Fig 3.3) [46]. As described before, there are two types of

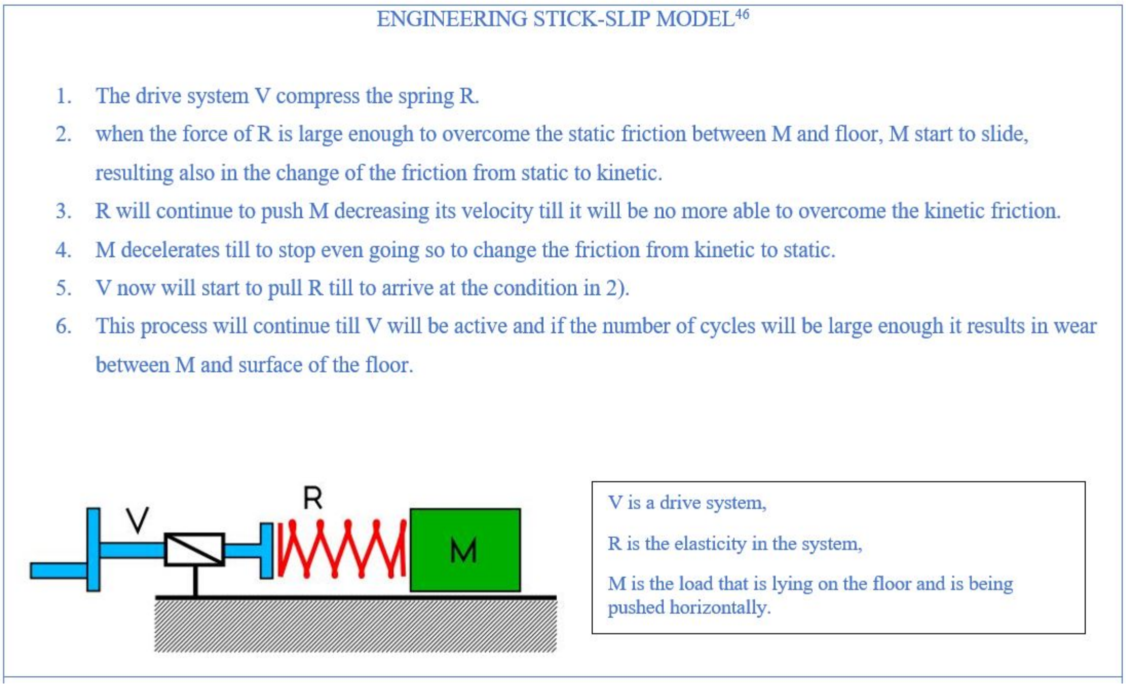


Figure 3.3: engineering stick-slip model

friction: static, caused by the molecular bonding that occurs when two surfaces are in contact, and kinetic, caused by surface roughness, which impedes the motion of the two bodies relative to each other. The stick slip-phenomenon occurs when two surfaces, alternate their contact condition between sticking to each other, due to static friction, and sliding over each other, with a corresponding change in the force of friction due to the passage from static to kinetic friction and vice versa[45].

The fretting regime

The fretting could be divided in regime using a correlation between dynamic tangential force and displacement measurements[38]. Here, there is a suggestion of division by Vingsbo and Soderberg in relation at the different type of damage presented on the surfaces[38]:

- Stick regime:
  - Surface damage: damage by wear and corrosion very limited
  - Crack formation: absent
- Mixed stick-slip regime.
  - Surface damage: Wear and oxidation effects are small.
  - Crack formation: Accelerated crack growth related to increase of the fretting fatigue.
- Gross slip regime.
  - Surface damage: Severe surface damage by oxidation-assisted wear, presence of fretting wear.

  - Crack formation: limited

- Reciprocating sliding regime.
  - Surface damage: damage by sliding wear and the wear rates become characteristic of unidirectional sliding.
  - Crack formation: limited.

Fretting test

Fretting tests are experimental way to determine the different tribological properties of a material under fretting condition, generally the more surface related one. These parameters are usually used for post analysis, for making comparison between different materials for a specific need. Usually, the scientists use different types of fretting testers commercially available for extracting data and use them for improving the tribology properties of the materials[47].

Factors affecting fretting

There are several factors affecting fretting behavior of a contact[39]:

- Contact load: influences the fretting condition, regulating the sticking during the contact, for that reason the wear rate it' s also linear dependent by it.
- Displacement: is the sliding amplitude
- Number of cycles: the value of it influences the presence of fretting wear and fretting fatigue. For small values there is a very small quantity of wear that is negligible, overcame that period there is one where the wear is constant, and then a period were the wear increase linearly.
- Temperature: influences the formation of the oxide layer, and consequently the wear mechanism.
- Relative humidity: an increase of it, helps to decrease the tribo-corrosion phenomenon.
- Inertness of materials[48]: this parameter depends material by material and it influences the formation of the oxide layer.
- Frequency[49]: its regulation influences the occurring time for the wear formation. it's suggested to increase its value when it's used a large value of displacement.

3.2.2 representation of fretting regimes

In their work Vingsbo, O. and Soderberg give a graphical representation of the regimes in the fretting map i.e. a graph that correlate different operating parameters of the phenomenon[38]. In these representations it must be said that the lines that indicate the limits between the different remiges are direct measurements of their work, while the reciprocating sliding limit is obtained by literature[38].

- FRETTING MAPS
  - Normal Load vs Displacement(Fig 3.4)[38]: the map show that there is a linear dependence of the boundary limits of the mixed stick slip regime. Taking this map in example we can assume that an high increase of normal load or displacement amplitude can avoid the fretting phenomenon[38]. The normal load imposes a pure sticking situation, while the displacement amplitude, increase the sliding condition, that usually gradually reduces the fretting fatigue till the phenomenon will be pure reciprocating sliding[38].

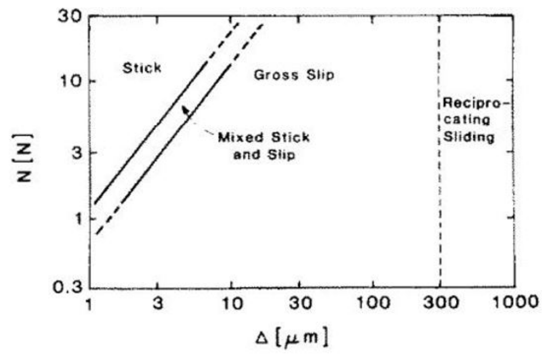


Figure 3.4: Fretting map N[N] & Δ[μm]

  - Normal Load vs Tangential Force(Fig 3.5)[38]: this representation shows a very strict range of values for the mixed stick slip regime. It's clear how the interdependence between normal load and tangential force is strongly connected with the contact conditions[38].

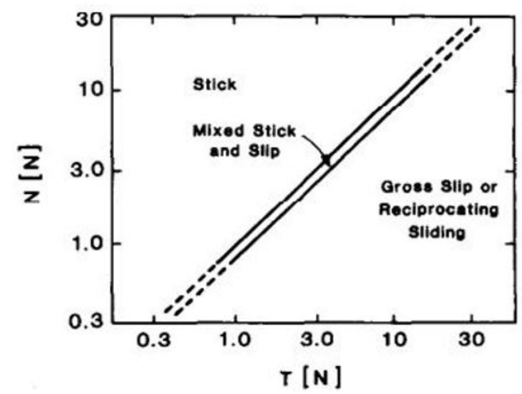


Figure 3.5: Fretting map N[N] & T[N]

  - Frequency vs Displacement(Fig 3.6)[38]: looking at this fretting map appears clear that the frequency has a more complex behavior. That's because it influences the mechanical properties of the material both through temperature effects and strain rate hardening effects[38].

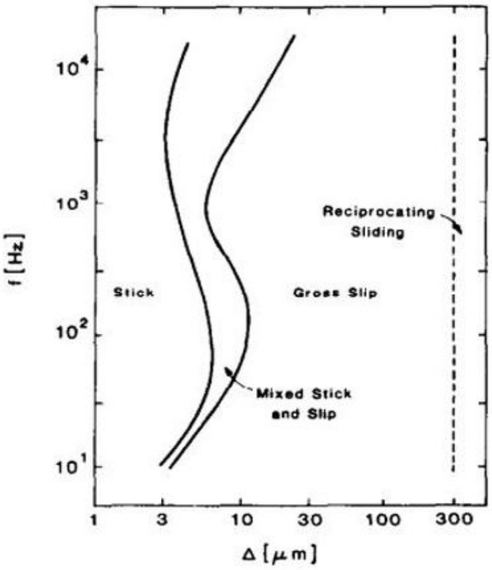


Figure 3.6: Fretting map f[Hz] & Δ[μm]^{X}

- FRETTING LOOPS The fretting loops (Fig 3.7) are a graphical representation of the fretting regime that is occurring[50]. Usually the shape of them is constructed dividing cycle by cycle, and putting in correlation, the tractional force (Q) with the applied displacement (Δ). In literature Q could be also the coefficient of friction, because the tractional force is dependent by it and by the Normal load[50].

  It's useful understand that (Δ) is the displacement applied and it includes all the elastic deformations in the system between the contact point and the position at which the displacement is measured i.e. a combination between contact, bulk specimen, fixture and rig elastic displacements[50]. This combination can be described by the stiffness of the system S, which role is to determinate the shape of the loop[50]. The actual contact slip amplitude ($\delta*$) is commonly determined by post-processing of the force and displacement data. It and the stiffness depend on the design of the test apparatus, but (Δ) also by the load applied[50].

  In this example, taken form the work of S. R. Pearson and P. H. Shipwa[50], are represented three different loops with the same applied displacement and the same stiffness of the system, but they have different shape and slip amplitude caused by different tangential forces applied or different normal load or different friction coefficient[50].

  Being a loop an hysteresis cycle, the area inside of it represents the energy dissipated by the fretting phenomenon in that cycle[50].

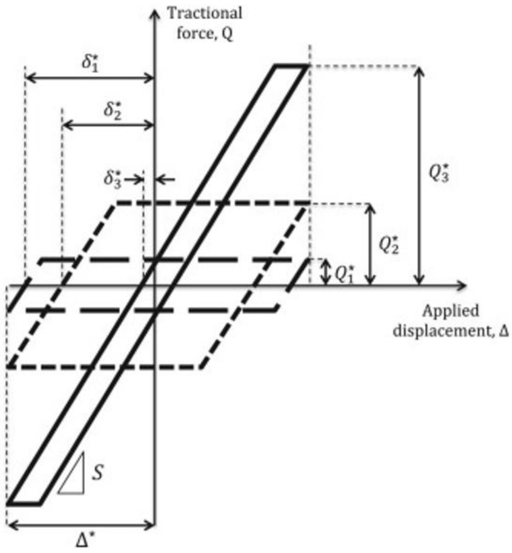


Figure 3.7: Fretting loops Y

3.2.3 Fretting derivation of wear coefficient in fretting

$$k_{ACHARD} = \frac{V^W}{4n\delta^* N}\left[\frac{m^3}{mN}\right]$$

$$k_{energy} = \frac{V^W}{E_d^{TOT}}\left[\frac{m^3}{J}\right]$$

V^W= Wear volume
n= Number of cycles
δ*= Slip amplitude
N = Normal Load
Ed= Energy dissipated in a single scycle

Figure 3.8: Wear coefficient equations

These two equations (Fig 3.8) are the two main ways to measure the wear coefficient of the fretting phenomenon. The first one is the Achard's coefficient and is the most used for the characterization of the materials, the second one is related to the energy dissipation, and it 's used nowadays by researchers for the tribological analysis[50]. The two equations are dimensionally equal and with the new methods, that analyze the coefficient of friction extracting directly the fretting loops, they are reliable ways to measure the wear data[50].

However, generally, the fretting loops are not measured as part of the experimental procedure. The only measurements available to the researchers are the applied displacement amplitude and either the normal load or the maximum tractional force[50].

Taking in consideration these limitations, the scientists usually make the approximation $\delta * \approx \Delta$ for calculating the k_{ACHARD}[50]. This assumption modifies the shape of the loops, but that is not a real problem because it allows anyway a good approximation of the reals one[50].

In some empirical experiences, where the friction coefficient was plotted in function of the slip amplitude, there were values of the wear volume that tend to zero even if the applied displacement is not zero[50]. This situation could be explained with a zero value of the $\delta *$, caused by the absence of movement, thanks to the elastic properties of the material, or because during the experiences was reached the time threshold of fretting (a time where there is no more wear associated to the fretting phenomenon)[50].

The limitation of the second wear coefficient is the existence of an energetic threshold, experimentally proved, that when overcome there is dissipation but no volume wear is observed[50].

3.3 Formulation of objectives and criteria

3.3.1 Purpose of this work

In relation to the fretting problem registered on the surfaces contact between the abutment and the fixture, the aim of this study was to evaluate the tribological behavior of three different types of titanium against zirconia, to show which of them had the best behaviour under fretting conditions.

The main interest was to find the titanium with the best friction value (the smallest) or the best non-sliding behavior (this because the sliding is related to the wear) in fretting condition against the Zirconia. This type of experiment was made according to the new increasing trend in dentistry, to realize new design of dental implants with prothesis + abutment in zirconia custom made[50], that usually interface with titanium fixture[7].

3.3.2 Literature survey

After a quick comprehension of the fretting mechanisms, for a formulation of a protocol, a literature survey on the main parameters and materials was performed on papers about the tribological behavior of dental material in fretting conditions. The keywords used were “dental implant fretting”, “fretting biomaterials”, “Params fretting dental implant test” and “Contact pressure dental implant (VM)”. The search engines on which the searches were made were Science Direct, Springer and Google Scholar.

Materials

The materials (Fig 3.9) found in the papers for a dental implant use can be grouped in:

- metallic biomaterials (commercially pure titanium, Ti-6Al-4V and other titanium-based alloys, Co-Cr-Mo, stainless steel)
- ceramic biomaterials (alumina, zirconia, glass-ceramic materials, steatite, silicon nitride, zirconia of the latest generation)
- polymers and composites (resin composites),
- Human bone tissue.

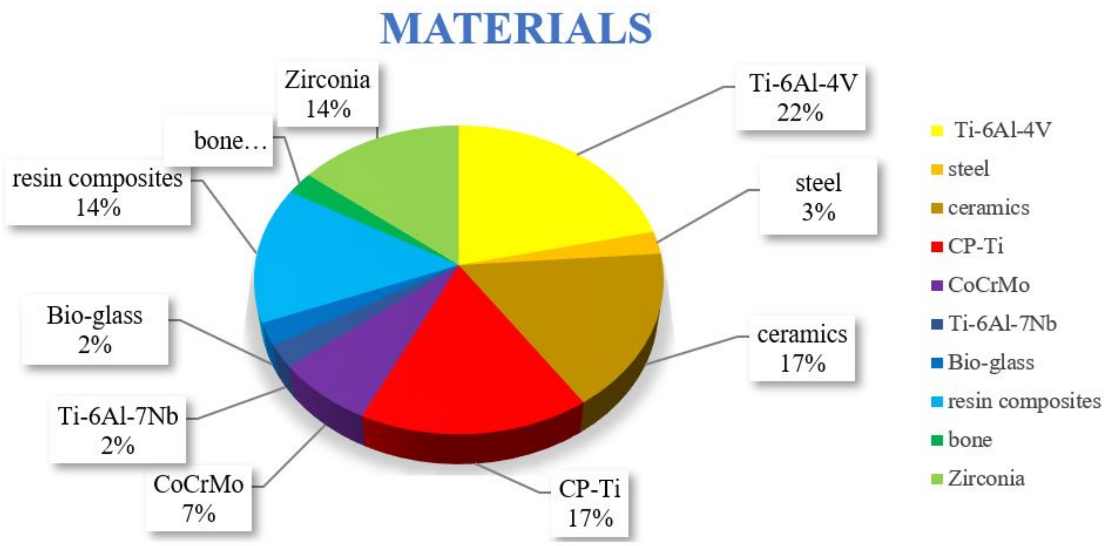


Figure 3.9: Materials tested in dental tribology papers

Counter materials

The counter materials in the analyzed paper present mostly a ball shape with a diameter between 8-10 mm, the others, less used, are in the pin on disk configuration.

Figure 3.10 reports the relative presence of the tested materials in the fretting experiences of the papers analyzed. Titanium materials are the most frequently used, followed by Zirconia and Resin composite.

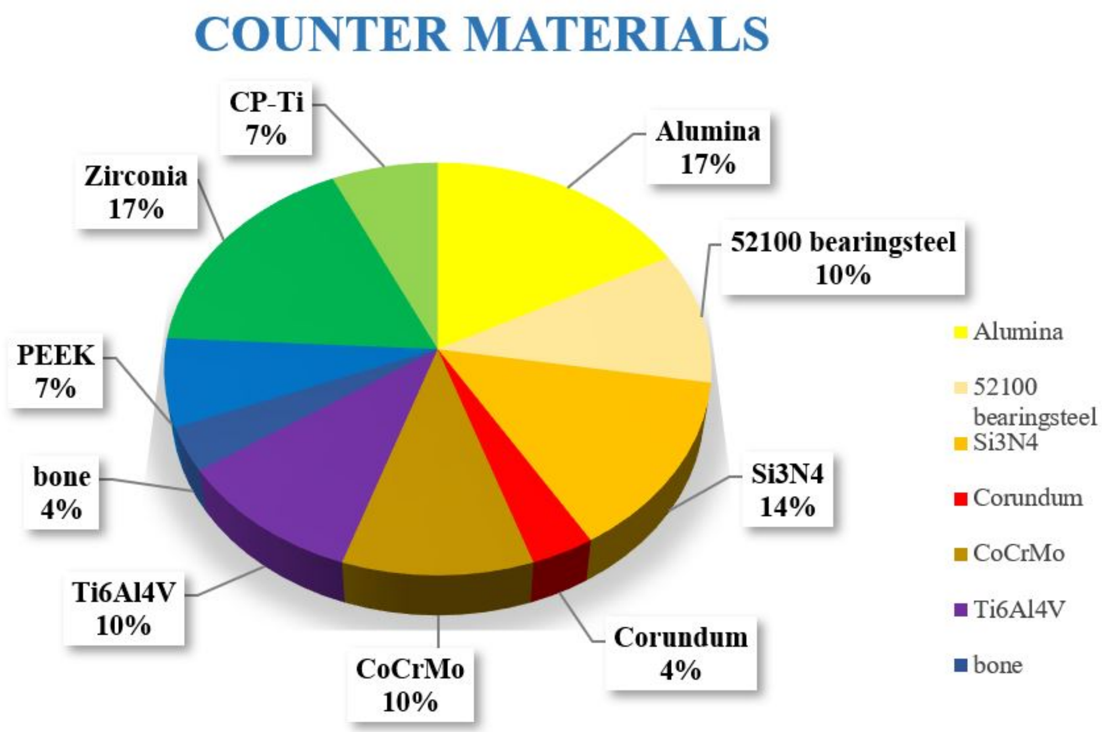


Figure 3.10: Counter materials tested in dental tribology papers

Lubricants

From the papers analyzed it' s clear how the clinicians test the materials mostly with the aid of lubricants (Fig 3.11). These last are predominantly different types of artificial saliva and, except for few cases, their use is related to reproduces the oral environment.

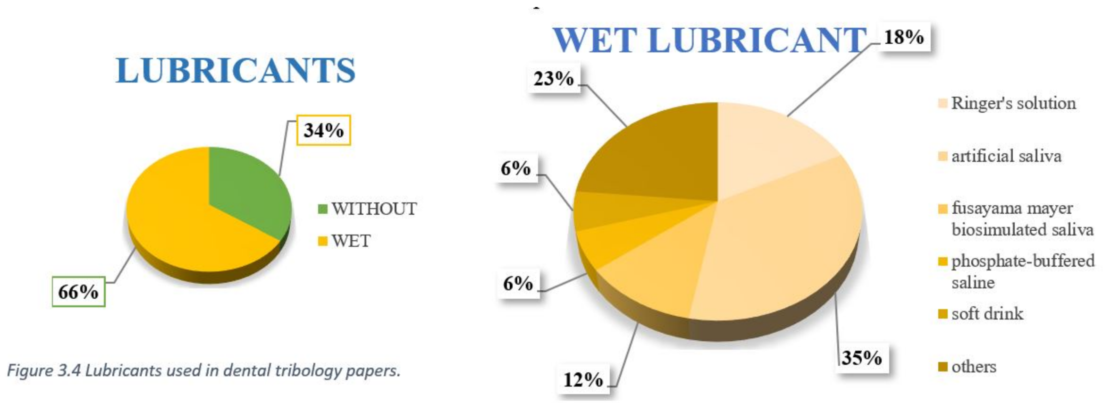


Figure 3.11: Lubricants used in dental tribology papers.

Instruments

In Figure 3.12 It easy to recognize that the instruments, used for these types of investigations, are several but that generally are different types of tribometers.

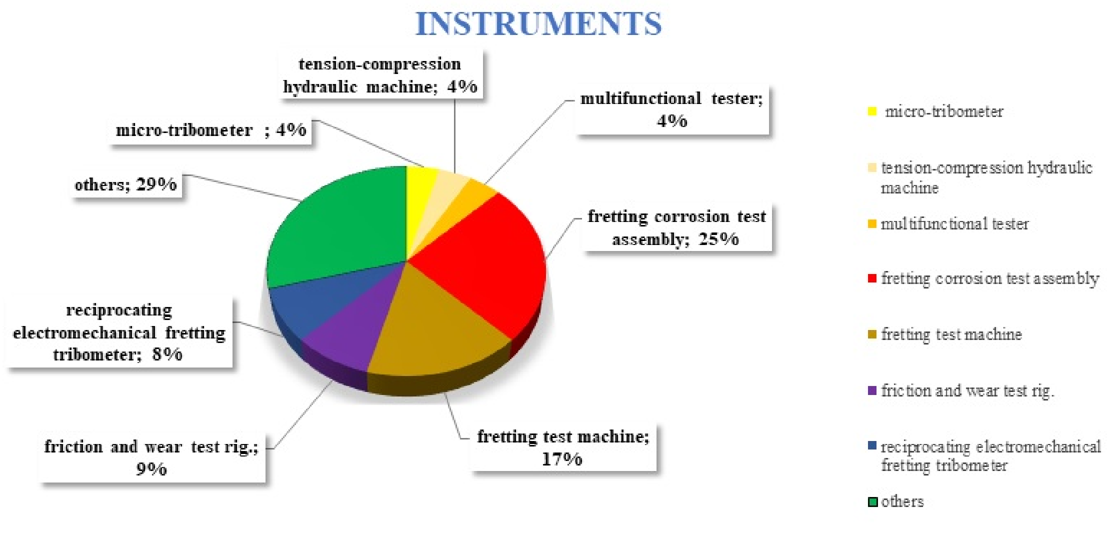


Figure 3.12: Instruments used in dental tribology papers.

Test parameters

The parameters found for the setting of the fretting condition are now listed: Number of cycles, Frequency, Normal load, Stroke Length. The data shown an high value of variance. That because the fretting phenomenon is a mix of different parameters and there are no clear guidelines for set them for having the expected results.

NUMBER OF CYCLES

N° CYCLES (n°) descriptive statistics	
Average	11233.33333
Standard Error	2563.672815
Median	10000
Mode	10000
Standard deviation	10876.74259
Sample variance	118303529.4

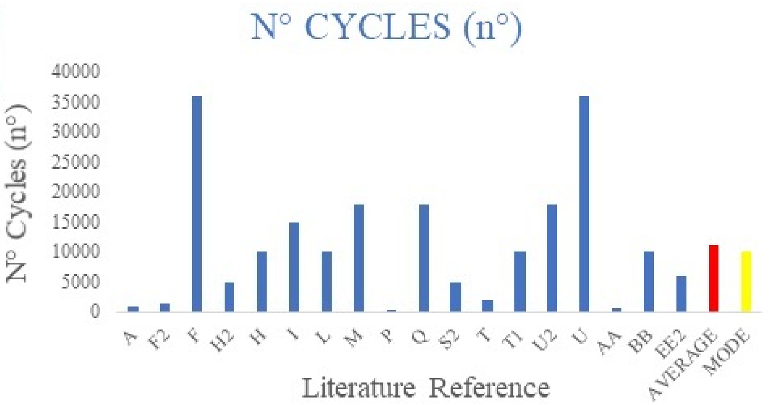


Figure 3.13: Number of Cycles used in dental tribology papers.

FREQUENCY

FREQUENCY (Hz) descriptive statistics	
Average	3.94047619
Standard Error	0.660290512
Median	4
Mode	5
Standard deviation	3.025831251
Sample variance	9.155654762

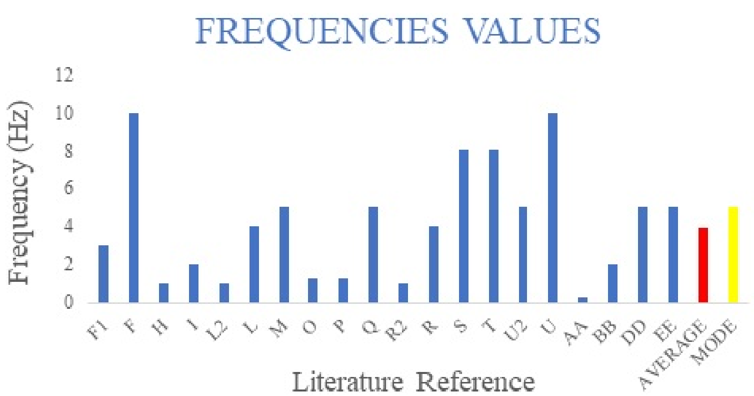


Figure 3.14: Frequencies used in dental tribology papers.

NORMAL LOAD

STROKE LENGHT (mm) descriptive statistics	
Average	0.192541667
Standard Error	0.037281148
Median	0.14
Mode	0.18
Standard deviation	0.182639578
Sample variance	0.033357216

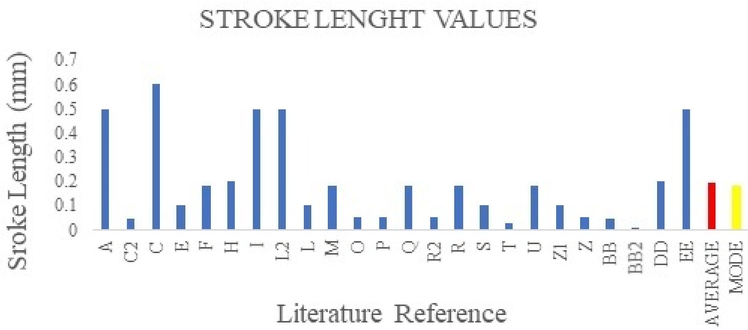


Figure 3.15: Number of Cycles used in dental tribology papers.

Temperature

In this graph the room temperature and the body temperature are classified only when explicitly declared in the paper. Under the label not known are classified the experiments where there are no indication about this parameter. Could be good assume that in these cases that the temperature is the same of the room

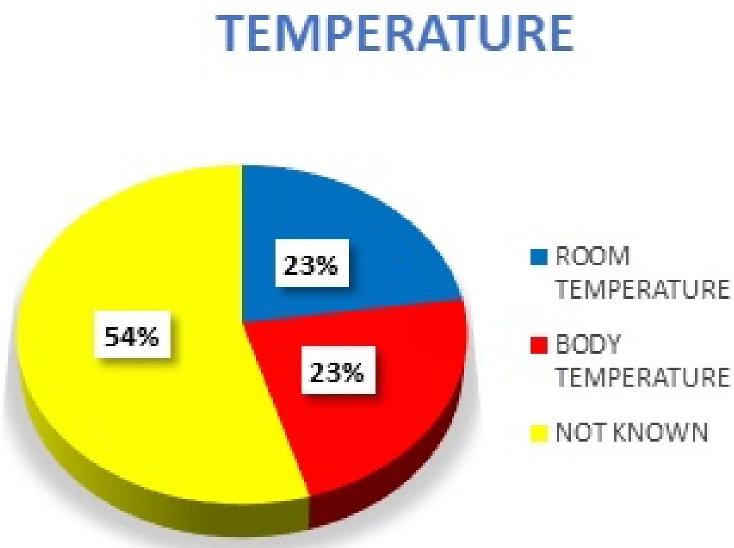


Figure 3.16: Temperatures used in dental tribology papers.

Summary

Here a summary of the operating parameters, in form of average ± standard deviation values, for a fretting experiment. The humidity was not reported because only in two papers was declared. Talking about the analysis method and results, is not easy to make a descriptive statistic of them because the methods are different paper by paper and the values of results are mostly represented with graphical methods, making it difficult to define exact values.

PARAMETERS	UNITS	VALUES
STROKE LENGHT	mm	0.19 ± 0.18
FREQUENCY	Hz	3.94 ± 3.03
N° CYCLES	n°	11233.33 ± 10876.74
NORMAL LOAD	N	5.90 ± 6.01
CONDITION		wet
TEMPERATURE	C°	Room temperature

Figure 3.17: Summary of the parameters used in dental tribology papers.

3.3.3 Definition of the protocols

In order to make a comparison of the different tribological behaviors of the materials selected, two different protocols were developed.The materials used for the experiments were(Fig 3.18):

- 9 Zirconia Balls
- 3 Ti-6Al-4V pins
- 3 DLC (Ti-6Al-4V) pins
- 3 CPTi pins
- Bioxtra as lubrican

Figure 3.18: Summary of the parameters used in dental tribology papers.

Zirconia balls

All the balls were made of zirconia (ZrO2) (CERATEC TECHNICAL CERAMICS BV, The Netherlands) with a diameter of 3 mm (Fig 3.19).

ZIRCONIA

Figure 3.19: Zirconia ball.

Zirconia was used for the first time in dentistry in 2004 for the construction of a crown and a bridge, and now are used also for the entire abutment crown complex. That because its high fracture toughness, strength, fatigue resistance and its good biocompatibility, permit to bear the masticatory load and to do not alter the oral enviroment[7, 28, 51, 52]. Zirconia is an oxide formed from a less pure material: the zircon. Its processing involves in the separation and removal of unwanted materials and impurities, some of which are also radioactive (characteristic to not be overlooked because the implants are generally long-term) [51, 53].

High purity products are obtained at low temperature, with a method that exploits the exothermic nature of the reaction between chlorine, minerals containing zirconia and coke. The mineral is heated in a melting pot covered with SiO_2 and with coke in order to transform all the oxygen into CO and CO_2[28]. At the same time, the chlorine is bubbled producing volatile chlorides including zirconylchloride, which will be then separated by the others. Finally, the product is purified and then calcinated at 1200C[28, 54].

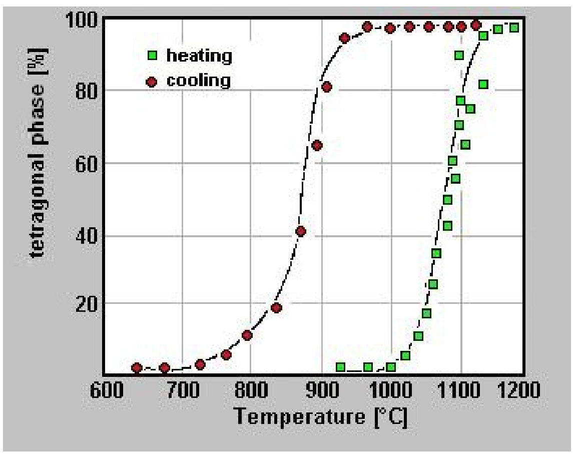


Figure 3.20: Zirconia transformation monoclinic to tetragonal and vice-versa.

The Zirconia is polymorphic: it exists in the monoclinic form from room temperature to 1100C, in the tetragonal form from 1100C to 2370C and in the cubic at higher temperatures[28, 51, 55].

The transformation from monoclinic into tetragonal Zirconia is accompanied by a great diminution in the structure volume (5%)[28]. Looking the productive process of Zirconia, this transformation occurs after the production, while during the cooling there is the reverse reaction, with a consequent expansion, that could lead to microcracks generation[28].

A solution for having a high purity in the final product but without cracks is that to use additives or stabilizers, such as CaO, MgO, and especially Y_2O_3, during the process[51, 52, 53]. This provision allows the formation of meta-stable Zirconia which, combined with processing variations, demonstrates exceptional ceramic properties and finds space in several application areas including the dental field[51]. Meta-stable means that trapped energy still exists within the material to drive it back to the monoclinic phase[51, 52, 53]. This type of configuration permits the stop of the cracks with an energy dissipation re-transformation of the grains, with also their volume expansion, from tetragonal to monoclinic (Fig 3.20)[51, 52, 53].

Main properties

The properties exhibited by the ceramic Zirconia depend on the degree, the type of stabilization and the treatment used during the processing, but in general this material has[52, 53]:

- High resistance
- High fracture toughness
- Excellent wear resistance
- High hardness
- Excellent chemical resistance
- High toughness
- Refractory
- Good conductor

All these characteristics are important for the dental prothesis because they permit to have a good aesthetic, a good biocompatibility, a good resistance at the chemical oral environment, a good dissipation of heat and a long life of service[51].

Now the Zirconia has seen an increment of its use, in form of Y-TZP, for dental purposes, thanks to the CAD-CAM technology that permit precise processing and custom made solutions[54]. One method mills the fully sintered block of Zirconia with no distortion (shrinkage) of the final structure. However, this type of implants undergo on fracture process because has a poor resistance to shear and tensile stresses. Indeed the failure rate of the implants,reported in literature, is around 4.1% after 1 year and 5.4% during the final torquing[25].

The Zirconia is very sensitive to imperfections or to the flaws created during the fabrication, therefore the application of supercritical loads culminate in immediate brittle fracture[25]. For that reason, the implants must be designed to have a good transmission of the masticatory force.If this requirement is not respected the implant will be subjected to internal stress and the crack propagation will not be stopped by the mechanism described before (Fig 3.21)[25, 55]. During the the thread design, all aspects treated before must be considered, because an excessive reduction in the diameter should also be avoided and the same attention must be paid during the

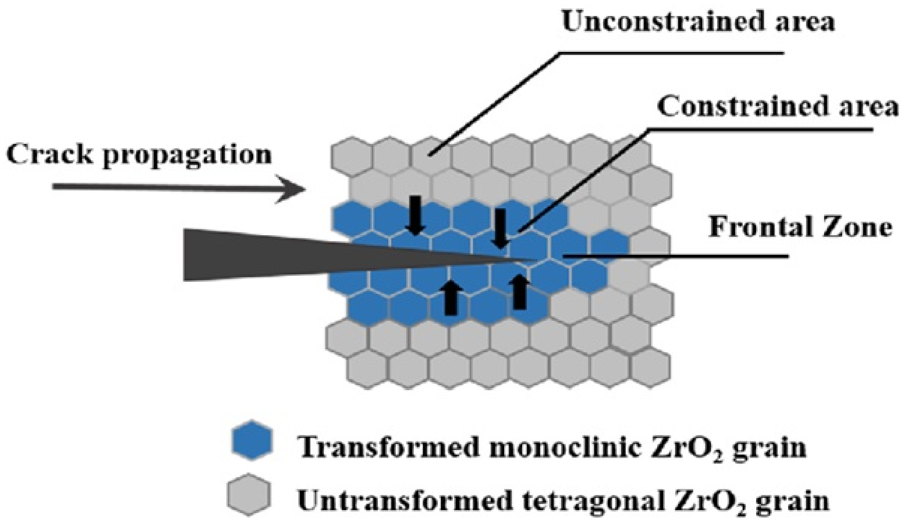


Figure 3.21: trasformation t to m during a crack propagation.

insertion of the implant for have not excessive bending moments[55].

The pins

Three pins(Fig 3.22) were made of commercial pure titanium (Acnis International, France), three in titanium alloy (Ti6Al4V) (Acnis International), and the last three titanium alloy (Ti6Al4V) coated with diamond-like carbon (DLC) (Vapor Technologies, USA).

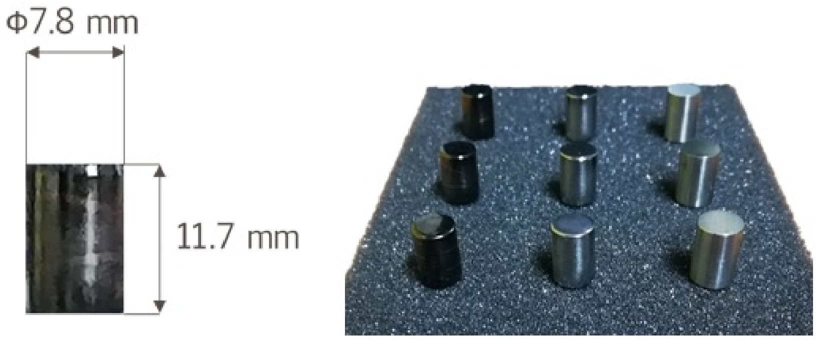


Figure 3.22: Ti-6Al-4V and DLC(Ti-6Al-4V) pins.

TITANIUM:

As concern the titanium its first application in dentistry was in 1977[24, 56]. That's because it has a long list of advantages respect to the other metallic biomaterials, it has; a better corrosion resistance, a low density, and lower elastic modulus (107-116 GPa), compared to stainless steels and cobalt alloys. This material making a comparison with the others metal based present a better mechanical properties but a worst tribological behaviour[24].

Main characteristics

- Biocompatibility: The biocompatibility of titanium is given by the fact that this metal is biochemically inert thanks to its passivation capacity, which makes it non-toxic[57].

- Passivation: Titanium has a very high resistance to corrosion, due to its ability to cover itself spontaneously with a layer of titanium dioxide, every time that it suffers mechanical damage with the presence of oxygen in the environment. The oxidation capacity of this element creates a surface passivation, which is fundamental for the corrosion resistance and the biocompatibility. In general, a good passivation treatment improves the abrasion behavior (a controlled layer of oxides is formed with a more regular morphology) [57].

- Radiopacity: A titanium prosthesis can be radio graphed in a clear way for understand better if the design is correct57.

- Gustatory neutrality: the passivating oxide layer inhibits the processes of electrogalvanic erosion and the consequent removes the more exposed particles of metal that usually annoy the patient.

- Thermal Conductivity: its thermal conductivity is very lower and so it doesn't promote thermal irritations phenomena at contact with the pulp[57].

Structure

At room temperature, titanium has a hexagonal close-packed structure (hcp) called phase . This structure remains stable up to 882 C, but above it the titanium structure undergoes an allotropic modification presenting a body-centered cubic structure (bcc) known as phase , which remains stable up to the melting point, at 166850 C24,55. As with other metals, in most cases, titanium is used as alloy, mainly for improve the wear resistance[24, 55]. The alloying elements play an important role in controlling the final microstructure and are commonly divided between:

- - stabilizers; the alloying substances come into solution preferably in the phase and increase its stability temperature. Among these elements are present: Al, O, N, Zn, Sn. In general, the phase promotes creep resistance, increasing resistance to deformation, but as consequence of that, it creates problems during the processing. These properties are related to the hexagonal structure, which has limited deformation capacity, allowing ductility only with a specific orientation. It is accompanied by good weldability[56].

- - stabilizers; the alloying substances come into solution preferably in the phase and decrease its stability temperature. Among these elements are present: vanadium, chromium, molybdenum, tungsten, tantalum, niobium. Phase reduces the resistance to deformation at high temperatures, even if all the mechanical properties strongly depend on the heat treatment[56].

ASTM F67 (Commercial pure titanium)

Being made up almost entirely of titanium, its structure is typically monophasic -type (HCP) with grains with a diameter from 10 to 150 mm, which sizes are based on the type of processing performed[24]. Its configuration is due to the presence of interstitial atoms (C, N, O) in its crystal lattice, which can produce a strengthening of the material. The presence of titanium oxide (TiO_2) on the metal surface increases corrosion resistance and contributes to a better biological impact (good osseointegration)[56, 57].

The ASMM (American Society for Testing and Materials) ranks this titanium in 4 different grades based on the impurities (mainly the oxygen percentage):

- Grade 1: oxygen content 0.18%. This quality has low tensile strength and high ductility.
- Grade 2: oxygen content 0.25%. it has a greater resistance than grade 1. It is the most widely used commercially pure titanium and offers the best compromise of strength, weldability and formability.
- Grade 3: oxygen content 0.35%. It has greater strength and lower ductility respect to the others before mentioned; more over it is well weldable.
- Grade 4: oxygen content 0.40%. and with higher resistance characteristics.

Designation	Tensile strength (min) MPa	ksi	0.2% yield strength (min) MPa	ksi	Impurity limits, wt% N (max)	C (max)	H (max)	Fe (max)	O (max)
Unalloyed grades									
ASTM Grade 1	240	35	170	25	0.03	0.10	0.015	0.20	0.18
ASTM Grade 2	340	50	280	40	0.03	0.10	0.015	0.30	0.25
ASTM Grade 3	450	65	380	55	0.05	0.10	0.015	0.30	0.35
ASTM Grade 4	550	80	480	70	0.05	0.10	0.015	0.50	0.40

Figure 3.23: Different grades of CPTi and their different composition.

For each of these groups the maximum content of nitrogen, carbon, hydrogen, oxygen and iron was defined, as well as the minimum values of some mechanical characteristics. The oxygen content must be checked carefully, because it has a major influence on the yield strength and fatigue strength(Fig. 3.23)

ASTM F136 (Ti-6Al-4V)

It is an - alloy at room temperature, and its microstructure depends on the mechanical processing and on the heat treatments undergone. Here listed how the different heat treatments can influence the microstructure of the F136 alloy (Fig. 3.24):

1. If the alloy is heated at temperatures between 700 and I 950 C (below the transition) its microstructure will be grains very fine (3-10 microns) with a dispersion of crystals at the grain boundaries of the phase primary[55, 56, 57].

2. If the alloy is heated above 975 C (i.e. in the thermodynamic stability field of phase, BCC) and then slowly cooled down to room temperature, a biphasic structure is produced where the phase HCP (rich of stabilizer Al) precipitates inside the grains of the matrix BCC, in the form of oriented lamellas or needles[55, 56, 57].

3. If is heated above 975 C but the cooling down is fast a fine microstructure is obtained, that due to non-diffusive (martensitic) solid state transformation[55, 56, 57].

4. A fourth class of microstructures is obtained through a sequence of thermo-chemical treatments that use hydrogen (stabilizer of phase) as a temporary alligating element, which gets down the - transformation temperature, making possible a eutectoid transformation with the phase nucleation from the low-temperature phase[55, 56, 57]. With this process a microstructure with better yield strength (974-1119 MPa),better ultimate breaking load (1025-1152 MPa), better fatigue strength (643-669 MPa) is obtained compared with the corresponding structure of the lamellar alloys[55, 56, 57].

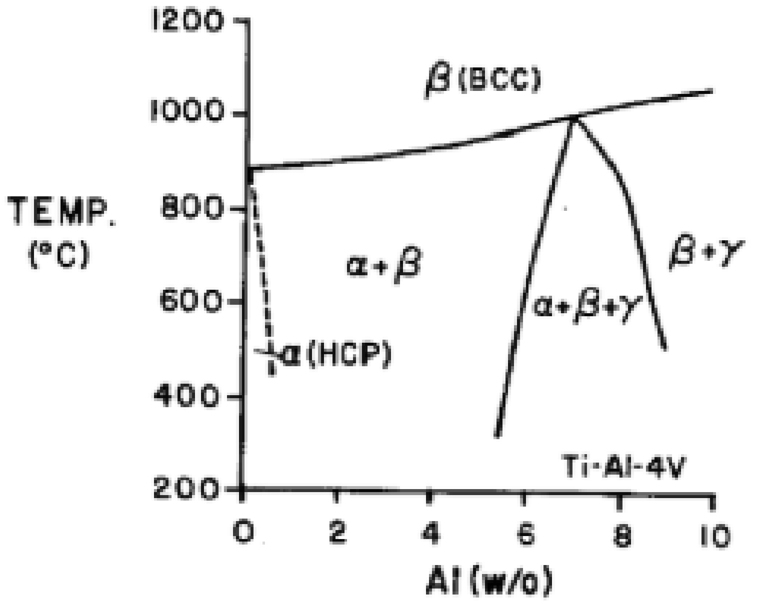


Figure 3.24: Ti-6Al-4V phase diagram.

In any case the mechanical properties depend on the quantity, size, shape and morphology of phase and on the interface density / , but for all the three main cases the difference is not so huge, except for the fatigue behavior, which seems better in the third case[56, 57].

The cracks start from the grain boundaries and from the interface / , when the material is stressed by cyclic loading regimes[57]. The mechanisms that govern the propagation and generation of the cracks are of different nature and are influenced by the type of microstructure[57]. The lamellar one shows a cracks propagation speed lower than the globular alloys, but is more subjected to the formation of the crack, the opposite behavior has been found for the globular with a smaller interfacial area[57]. When strengthening is needed, they are rapidly cooled and subjected to an aging treatment increasing the strength of a 30-50% and resulting also in a better fatigue strength[56].

The Ti-6Al-4V is very suitable for application that require contact with the bone, thanks to its biocompatibility, lower elastic modulus and the passivation mechanism[56]. However, there are concerns about possible health hazards brought by aluminum and vanadium, the first related to neurotoxicity and the second to citotoxicity[24, 57].

DLC

The Diamond-like Carbon is a form of amorphous (non-crystalline) carbon that can only be obtained as a thin film from carbon[58]. Carbon is an element presents in nature in three forms, sp1, sp2, sp3, different in how the atoms bond to each others[58]. The DLC consists of a structure in which the bonds between atoms are mixed, both sp2 and sp3[58](Fig 3.25). The prevalence of one fraction over the other determines the microscopic properties and the related uses in form of film: a prevalence of sp2 bonds gives the film a graphitic character, while a prevalence of the sp3 characterizes the properties more like diamond and guarantees better mechanical performances[58]. It is possible to produce the DLC either with PVD or CVD techniques, obtaining microstructures with very different characters and chemistries[58].

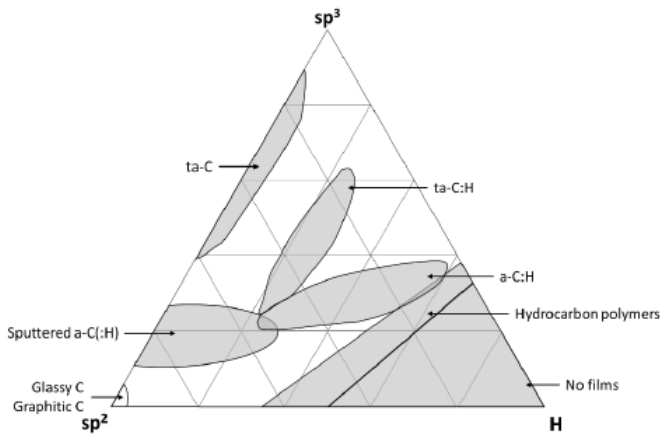


Figure 3.25: Ternary phase diagram for DLC formation with respect to sp2, sp3 and hydrogen content[C]

They are available[58]:

- pure carbon films, such as a-C (amorphous carbon) and i-C (tetrahedral amorphous carbon, with a higher content of sp3 bonds), with extreme hardness;
- hydrogenated carbon film, abbreviated a-C: H or ta-C: H, very versatile form in the properties;
- doped films with metals (titanium, chromium) and non-metallic elements (silicon), called a-C: Me and a-C: X, for specific functional applications.

Main properties

- High hardness: in some form similar at the diamond[58]
- Excellent resistance to wear and scratching[58]
- Low coefficient of friction: in some forms similar at the teflon[58].
- High chemical inertness, especially against oxidizing acids[58].
- High density that means also high impermeability to gas[58]
- Modular electrical properties based on the structure's chemistry[58].

Amorphous DLC (ta-C, a-C)

The amorphous DLC is formed by small groups of 5-10 nm of carbon in sp3 form of single or polycrystalline diamond, inserted in an amorphous matrix[59, 60]. Given the large percentage of carbon atoms, there is therefore a low percentage of hydrogen[59, 60]. Being hydrogen-free films, they are very hard and with an high internal stress[59, 60]. The tetrahedral amorphous carbon (ta-C) has a high fraction of sp3 bound and low sp2 atoms[59, 60]. The film is generally produced by the technique of sputtering against a solid piece of carbon[59, 60].

DLC (a-C:H or H-DLC)

This form of carbon has a structure mostly made up of sp2-bonded atoms with some sp3, containing hydrogen up to a maximum of 50% [59, 60]. This hydrogen is believed to aid the formation of the sp3 bond and therefore the percentage influences the relationship between the hybrid forms[59, 60]. These coatings are relatively soft, with relatively low friction coefficients and can be deposited in thicker layers[59, 60]. Generally the film is produced from a gaseous hydrocarbon source by PVD[59, 60]. It is usually referred to as a-C:H or H-DLC.

Lubricant

The lubrication of the contact surface of every pin was performed with BIOXTRA spray gel (Biopharm Srl, Italy)(Fig. 3.26). The BioXtra spray is a gel used for oral care in case of dry mouth[61]. its formulation mimics the Salivary Peroxidase System and other natural salivary components, to help the functioning of natural anti-bacterial non-immune and immune mechanism of the mouthcite[61].

The biofilm bacteria released from the pharma interacts with the mucosal surfaces in order to make them more accessible to the antimicrobial effects of the Lactoperoxidase System, Lactoferrin and Lysozyme[61].

Moreover, Immunoglobulins present in the Colostrum Extract also prevent the re-adhesion of the bacteria, already isolated by the pharma, to the epithelial cells with the consequent regeneration of the damaged mucosa[61]. This lubricant was used principally for replicate the lubricated oral condition and secondary to mimics the physiological environment of the mouth, thanks to the contained enzymes in the formula. This last characteristic remains a limit cause the not specific replication of the saliva's components.

Instruments

DUCOM MICROFORCE STATION

All the experiments were conducted by means of a pin-on-disk biotribometer (MicroForce, Ducom) (Fig.3.27) in combination with a moving stage. The instrument measures the frictional force, the normal force, and the coefficient of friction. The normal load (along the y axis) could be imposed through the Microforce software in a range between 0.01 and 10 N. However, the movement of the stage (along the x axis) could be imposed with the stage software with two different parameters: the frequency in a range that start from 1 Hz to 5 Hz and the stroke length in a range between 0.01 and 5 mm. The sliding movement of the stage is mono directional and perpendicular to the force sensor. The normal force and friction force are measured indirectly, thanks to a spring system of known stiffness. The elastic sheet inside are displaced along the x and y axes and their deformations are functional to calculate the two force, which are calculated through a formula that consider the values of two capacitors sensors, one for axis, and of the deformation constants of the springs. An accurate contact and the normal load, between the specimen and the surface on the moving stage, are reached thanks a piezoelectric actuator, that use as feed-back system, and the spring system along the y axis. The three parameters measured were video displayed in real time and saved with a tdms extension by the Microforce software.

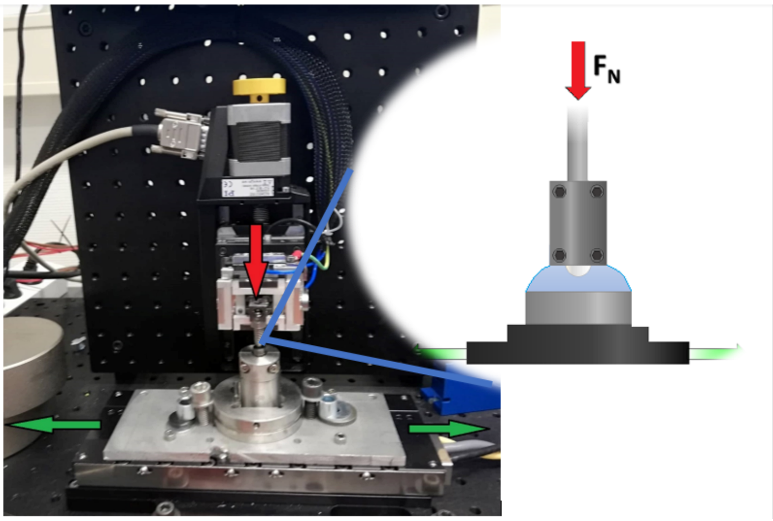


Figure 3.27: Ducom Microforce Biotribometer.

OPTICAL MICROSCOPE

The optical microscope (Fig. 3.28) was used to evaluate the presence and the extent of the wear scars. The magnification used in the experience is 10X, chosen by the three available (4X,10X,40X). All the captures were showed, modified and saved on the PC through the AMscope software.

LIGHT PROFILOMETER

The investigation of the fretting scars is in the μm scale, for this reason their analysis require the use of a light profilometer (profilm 3D Filmetrics) (Fig 3.29), since it can guarantee more precise measurements (accuracy below the nanometer can be achieved). The profilometer used, can generate three different types of light beam (white, colored and their mix). The beam is split in two: one half is reflected by the surface undergone the measurement and the other half is reflected by a reference mirror placed at a fixed distance from the beam splitter. The analyzing technique exploits the interference phenomena for generating light and dark bands (that form the interferogram) that are then used for calculating the distance from the material surface to the reference mirror, in terms of fractions of the wavelength. The captures of the surface of every pin are displayed on screen through the Filmetric software and post processed also with the online tools of Filmonline site.

Protocols

The protocols developed were thought for discovering the main tribological characteristics of the materials. First of all, there was an investigation about the main mechanical properties(Fig. 3.30) of the three different materials, here a summary of them.

After that a first protocol (Fig. 3.31) was outlined: The total time of work was 40 minutes divided in 3 step with different stroke length, 5 minutes with 0.02 mm, 5 minutes with 0.05 mm, 30 minutes with 0.1 mm. The protocol was thought with 1 Hz of frequency, with 0.5 N of normal load applied and in lubricated condition with BioXtra.

A second protocol (Fig. 3.32) was outlined cause the lack of appreciable results:

The total time of work was 30 minutes divided in 3 steps with different stroke length, 5 minutes with 0.02 mm, 5 minutes with 0.05 mm, 40 minutes with 0.1 mm. The protocol was thought with 1 Hz of frequency, with 2 N of normal load applied and in lubricated condition with BioXtra.

Before to make the test the following procedure and verifies were done:

Evaluation Of Hertzian parameters

The Hertzian parameters (Fig. 3.33) are important in this study, because the normal load applied is an operating parameter, but what is important in fretting is how this force is distributed and where. Analyze these two aspects is part of the understanding process of the load carrying capacity, of the friction, of the fretting wear and of fretting fatigue mechanisms of the materials under observation[**62.63**]. The Friction, as the other parameters before cited, is influenced by the roughness of the materials in contact and, responding to the two previous questions, the distribution of the force influences the asperities penetration and consequently the formation of "welding bridges"[**62.63**]. Hertz proposed a model to describe the stress and the deflections due to the contact between two elastic solids. He made the following assumptions for elaborate the solutions of the contact problems[**62.63**]:

- The strains are small and within the elastic limit.
- Each body can be considered an elastic half-space, i.e. the area of contact is much smaller than the characteristic radius of the body.
- The surfaces are continuous and non-conforming.
- The bodies are in frictionless contact.

The formulas change based on the geometry of the two solids in contact and were not treated in this essay, but we are referring for our calculations at the sphere on plane case[62, 63].

Hertzian parameters were then calculated for all the couplings between the pins of the three different materials against the zirconia balls for the two different normal load applied.

The circular contact area diameter(Fig. 3.34) is important to know because the materials will undergo on fretting condition only if the stroke length chosen is longer then half of the contact diameter. The data show that this condition is satisfied.

The maximum Hertzian contact pressure (Fig. 3.35) is the value of pressure exercised on the contact area when the normal load is applied. It is fundamental to understand if the pressure value is adequate for having fretting (a value too high conduct to the pure stick condition vice versa a value too low can conduct to the reciprocating sliding condition). Having no data about the contact pressure of the experiments in literature, the Von Mises stress between abutment and implant in a condition of preload od 100 N on the crown was taken as reference(Fig. 3.36). This last value is the average of the extracted values from the literature survey conducted on the topic " Von Mises value between implant and abutment"

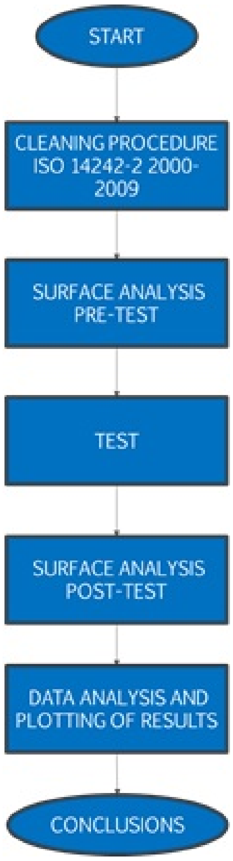


Figure 3.37: workflow of experimental procedures.

Procedures (Fig. 3.37)

for every test conducted the first step was the cleaning of the pins following the ISO 14242-2 2000-2009 procedure but using only demi water.

After that, it 's been proceeded with the surface pre-test analysis, making acquisition of the pins surfaces by mean of the optical microscope and the interferometer (with the white light technique). Same procedure for the surface of the balls through the optical microscope (the reason of that is because the interferometer analysis need of flat surface for give relevant results).

Then was evaluated the average profile and the roughness of the materials. Finished this procedure, the stage was prepared placing the specimens in the holders and lubricating the surface of the pins in contact with balls (Fig. 3.38).

Then the test was performed following the operational parameters of the protocol. Passed the prescribed number of minutes, the same procedures for the pre-analysis were actuated for the post analysis, but before o that, the surfaces of the specimens were cleaned with ethanol. Acquired the images, the data were processed and plotted and then some conclusions were drown up.

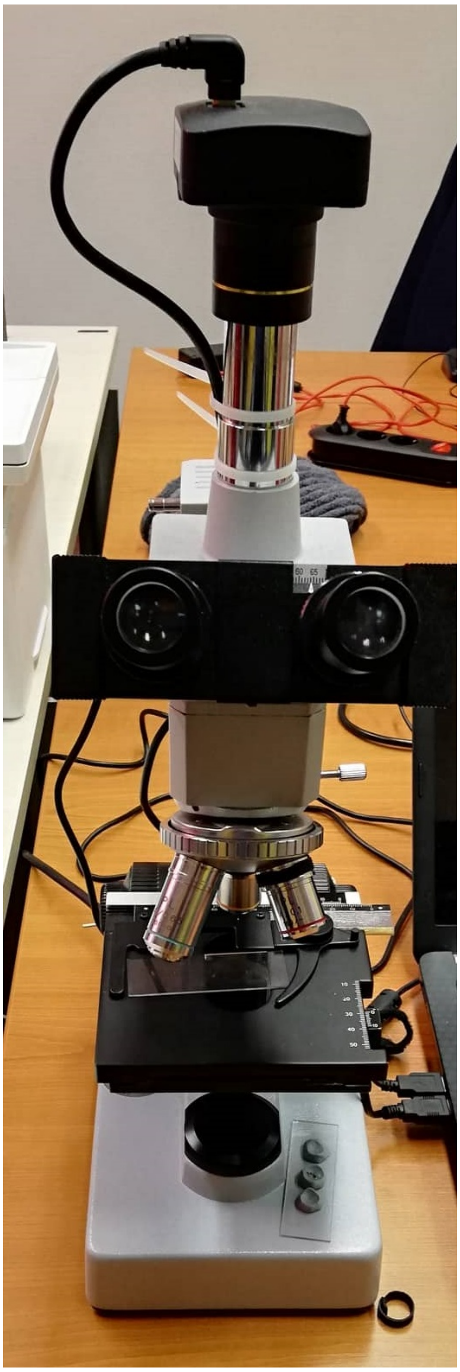


Figure 3.28: Optical Microscope.

Figure 3.29: light profilometer (profilm 3D Filmetrics)

	Elastic Modulus (E [Gpa])	Poisson Ration (v)	Hardness (HV [Gpa])
Zirconia	200	0.23	12
CP TI	110	0.37	4
TI-6Al-4V	113.4	0.342	1.15
TI-6Al-4V coated DLC	165	0.22	22

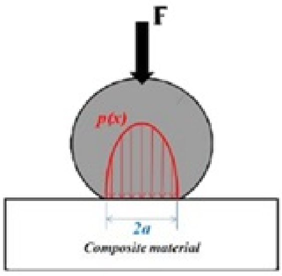
Figure 3.30: mechanical characteristics of the materials)

TOTAL TIME = 40 min Continuos	TIME STEP 1 = 5 min	TIME STEP 2 = 5 min	TIME STEP 3 = 30 min
NORMAL LOAD (N)	0.5	0.5	0.5
STROKE LENGTH (mm)	0.02	0.05	0.1
FREQUENCY (Hz)	1	1	1
CONDITION	WET	WET	WET

Figure 3.31: protocol 1

TOTAL TIME = 40 min Continuos	TIME STEP 1 = 5 min	TIME STEP 2 = 5 min	TIME STEP 3 = 60 min
NORMAL LOAD (N)	2	2	2
STROKE LENGTH (mm)	0.02	0.05	0.1
FREQUENCY (Hz)	1	1	1
CONDITION	WET	WET	WET

Figure 3.32: protocol 2



Contact radius (r) = $\sqrt[3]{\frac{3F}{8} * \frac{(1-v_1^2)\frac{1}{E1}+(1-v_2^2)\frac{1}{E2}}{1/D1}}$

Maximum pressure (p_{max}) = $\frac{3F}{2\pi a^2}$

r = contact radius
F = normal force
E_1 & E_2= elastic modulus of the two materials
v_1 & v_2 = Poisson's ratio of the two materials
D_1 = sphere diameter

Figure 3.33: Hertzian contact model and formulas

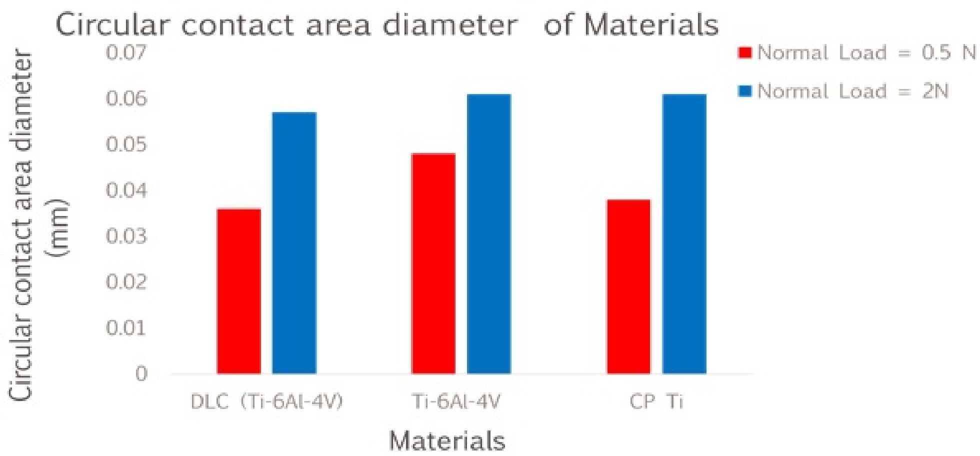


Figure 3.34: Hertzian circular area diameter of the materials used

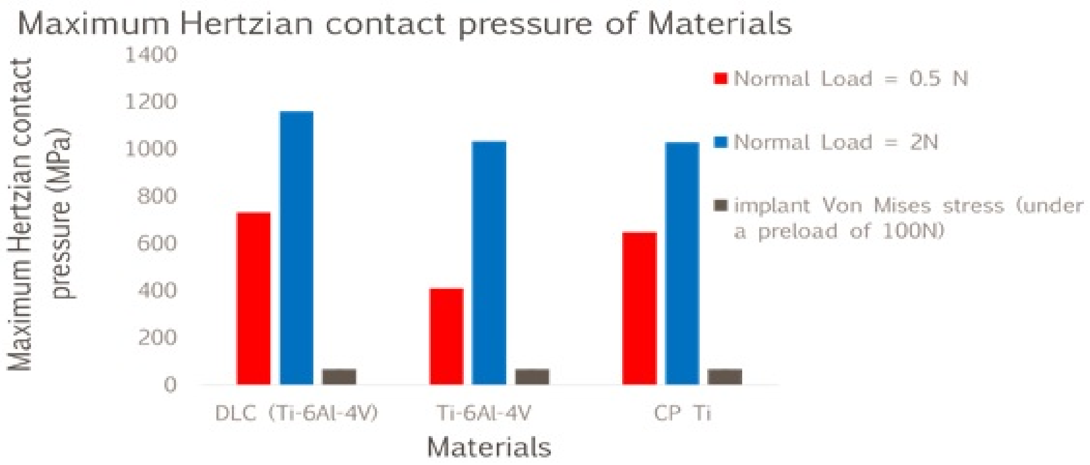


Figure 3.35: Hertzian contact pressure of the materials used

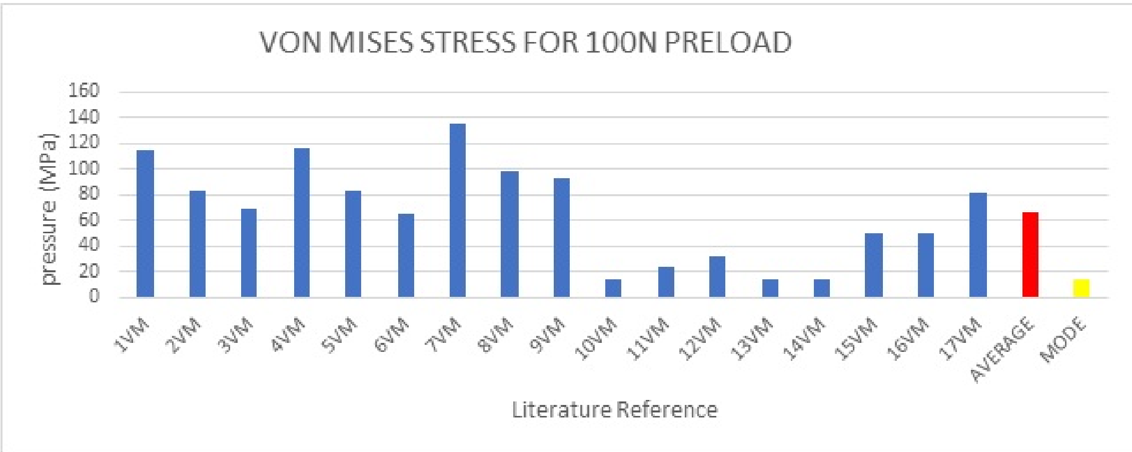


Figure 3.36: Literature survey of Von Mises values between implant and abutment

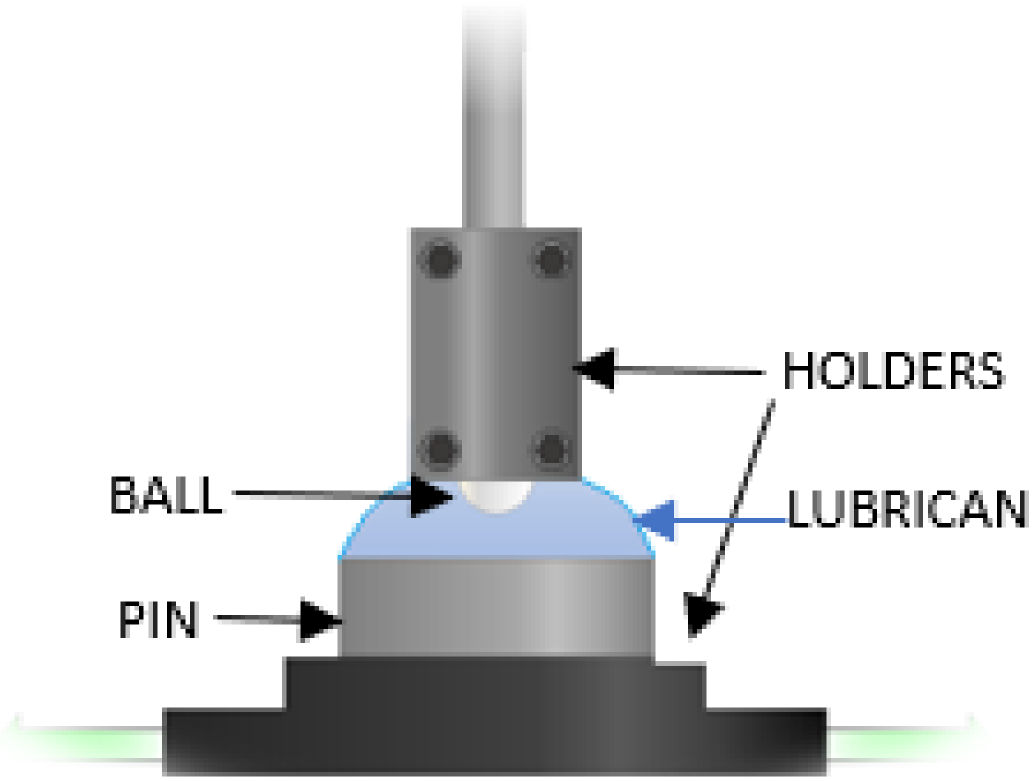


Figure 3.38: experiment preparation

Chapter 4

Results

4.1 Surface analysis post-test

4.1.1 Optical microscope analysis protocol 1

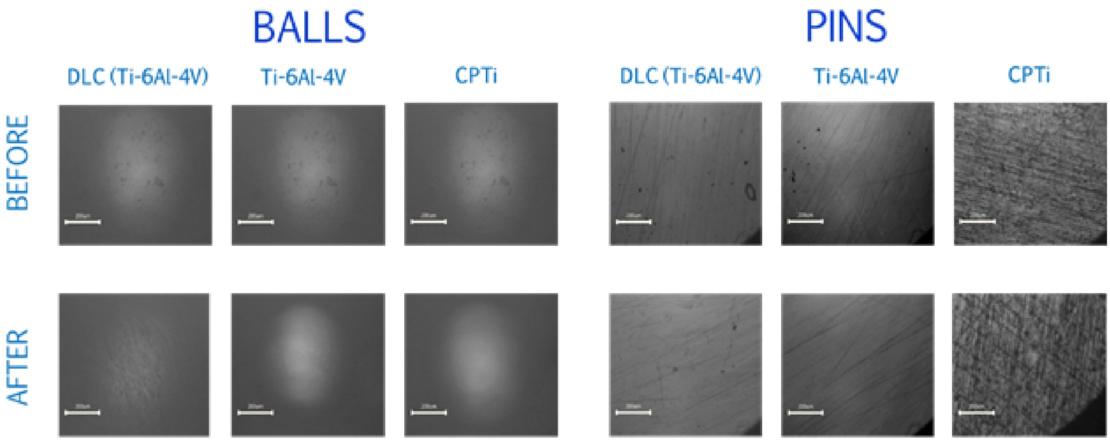


Figure 4.1: optical microscope comparison between before and after the test 1

In the figure 4.1 is shown an optical microscope comparison between before and after the test 1 of the different pins materials under investigation. Every photo used is representative for all the tests made with the same material (one for three tests). It's clear how there are no significant differences, only in the CPTi there is a slight discoloration on the contact area.

4.1.2 Interferometer analysis protocol 1

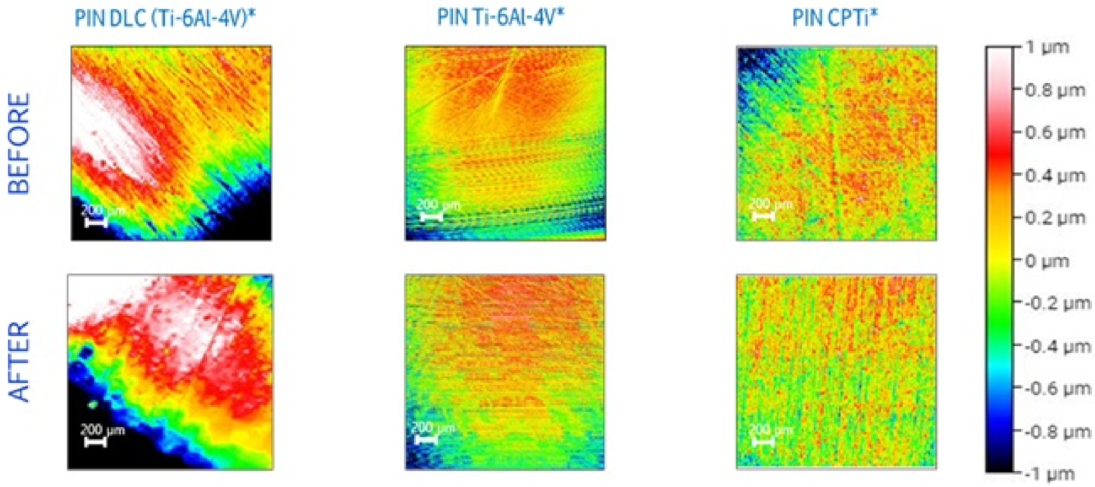


Figure 4.2: interferometer comparison between before and after the test 1

In the figure 4.2 is shown an interferometer comparison between before and after the test 1 of the different pins materials under investigation. The deep of the asperities is quite similar in every case, but the morphology is different because the orientation and the inclination of the pins was not the same in all the measures (cause the impossibility to have fix reference at the microscale). The discolorations on the CPTi pins surfaces was not perceived by the instrument.

4.1.3 roughness analysis protocol 1

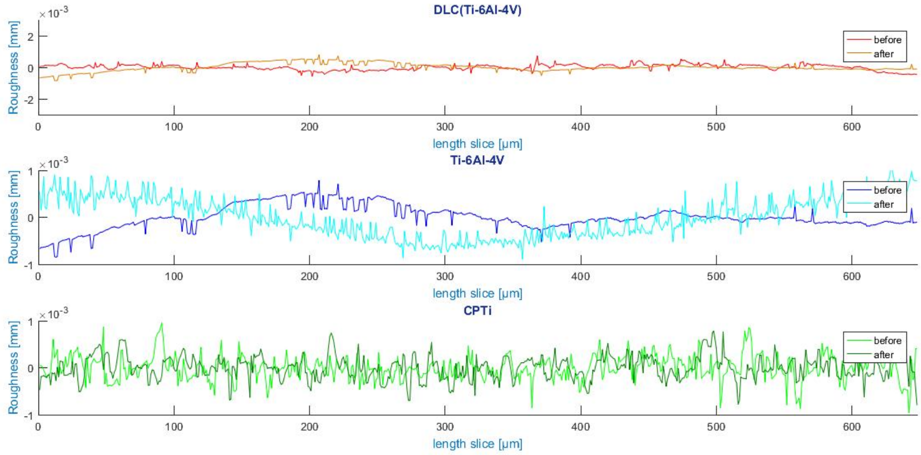


Figure 4.3: profile comparison between before and after the test 1

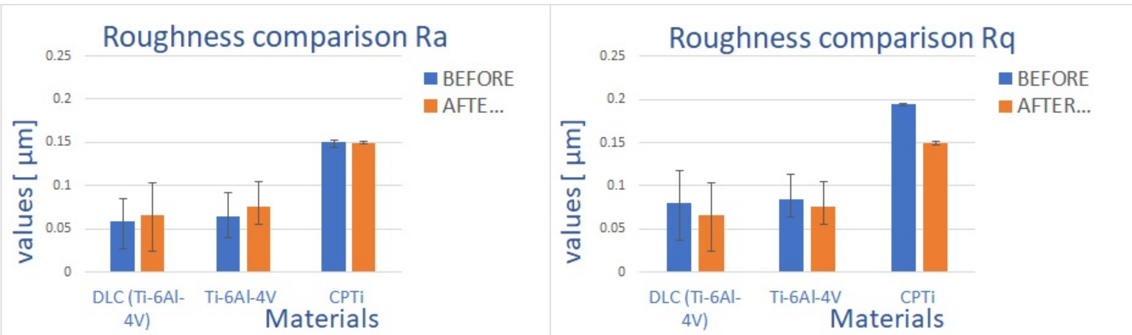


Figure 4.4: roughness values comparison between before and after the test 1, ISO 4287 R_a and R_q

The figures 4.3 and 4.4 show the roughness analysis and the surface profile of the pins. The extraction of the data was performed using a profilometer for analyze the surfaces. Talking about the profiles, they are a 2D representation of the asperities on the middle line of the pins surfaces (one image is representative for all the pins of the same material). Making a comparison between after and before the test we can notice that there are no remarkable changes. To confirm it we can look at the bar diagrams, where are shown the R_a and R_q ISO 4287 values of the materials (every bar is the average value of the R_a and R_q of the three different pins of the same materials).

4.1.4 Optical microscope analysis protocol 2

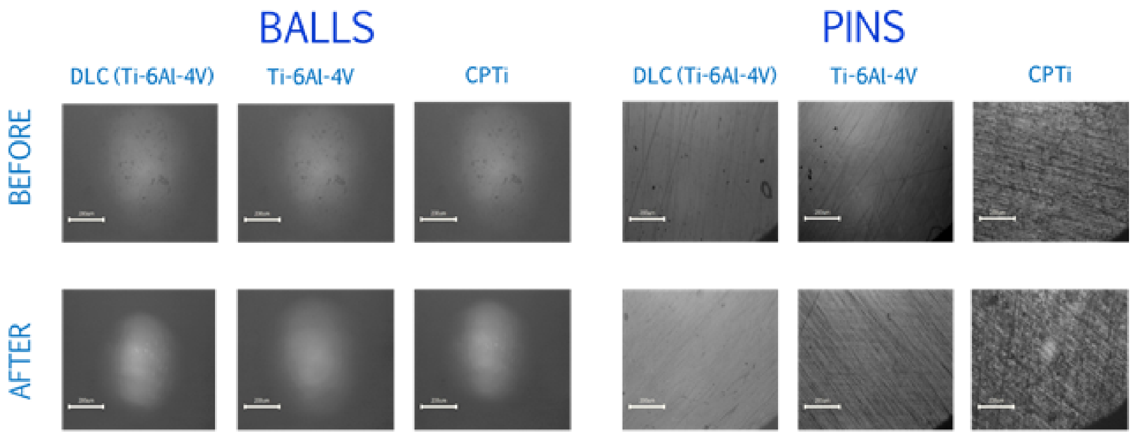


Figure 4.5: optical microscope comparison between before and after the test 2

In the figure 4.5 is shown an optical microscope comparison between before and after the test 2 of the different pins materials under investigation. Every photo used is representative for all the tests made with the same material (one for three tests). Making a comparison with the test 1 the results are similar for the DLC (Ti-6Al-4V) and for the Ti-6Al-4V pins while for the CPTi pins the discoloration on the contact area appears more marked.

4.1.5 Interferometer analysis protocol 2

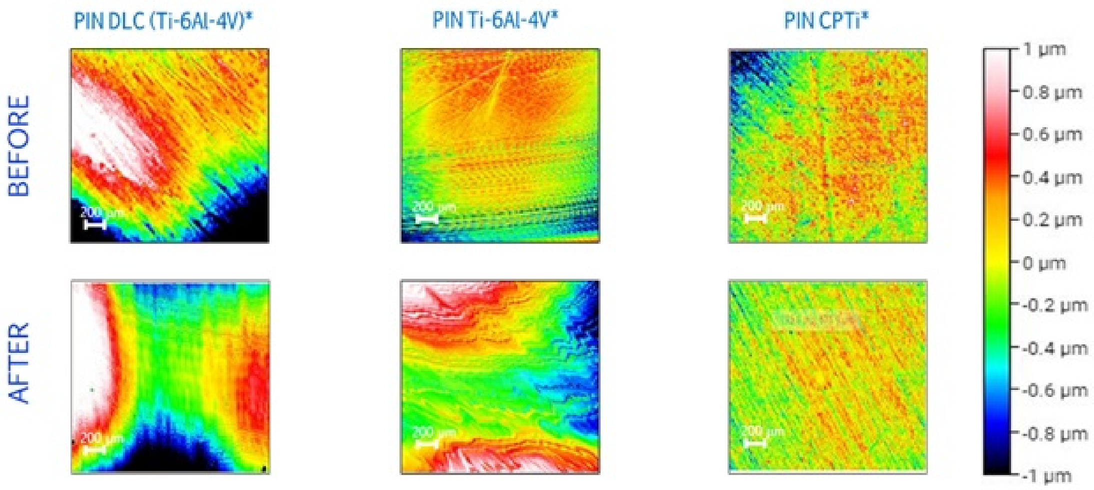


Figure 4.6: interferometer comparison between before and after the test 2

In the figure 4.6 is shown an interferometer comparison between before and after the test 2 of the different pins materials under investigation. The deep of the asperities is the same in every case, but the morphology is different because the orientation and the inclination of the pins was not the same in all the measures (cause the impossibility to have fix reference at the microscale). The discoloration on the CPTi pins was not perceived by the instrument.

4.1.6 roughness analysis protocol 2

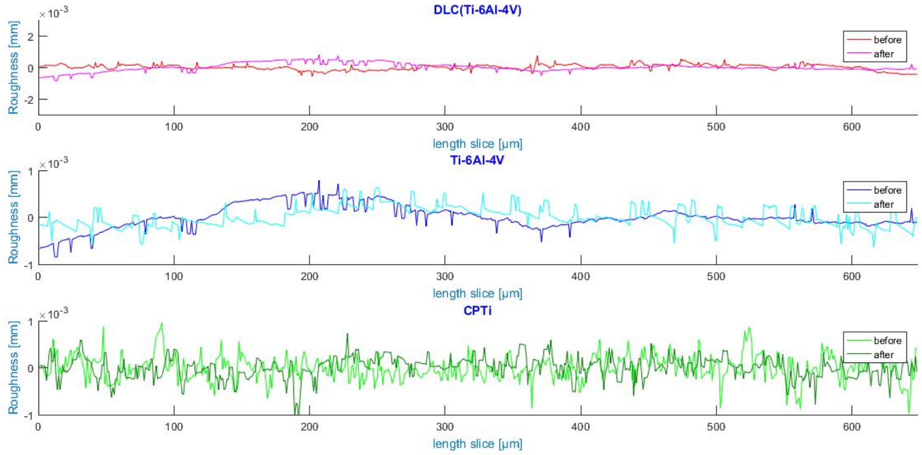


Figure 4.7: profile comparison between before and after the test 2

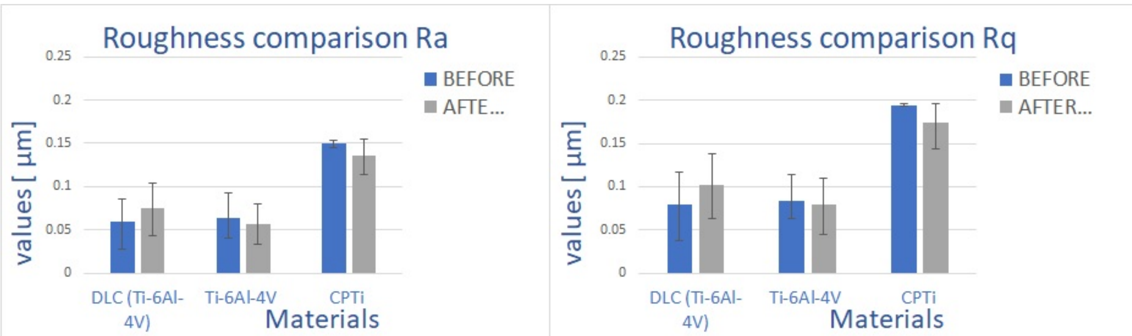


Figure 4.8: roughness values comparison between before and after the test 2, ISO 4287 R_a and R_q

The figures 4.7 and 4.8 show the roughness analysis and the surface profile of the pins. The extraction of the data was performed using a profilometer for analyze the surfaces. Talking about the profiles, they are a 2D representation of the asperities on the middle line of the pins surfaces (one image is representative for all the pins of the same material). Making a comparison with the protocol 1, we can see that the differences are more related to the measure of different area of the pins surfaces than to a real change of the values. The discoloration on CPTi pins surfaces continues to be irrelevant in the roughness analysis. To confirm it we can look at the bar diagram, where are shown the $_a$ and R_q ISO 4287 values of the materials (aevery bar is the average value of the R_a and R_q of the three different pins of the same materials).

4.2 Data analysis post-test

The data were analyzed through the MATLAB software.

4.2.1 Normal force analysis

Protocol 1

The normal force applied was 0.5 N and is percentage error was under the 5%, therefore making the parameter reliable (Fig. 4.9).

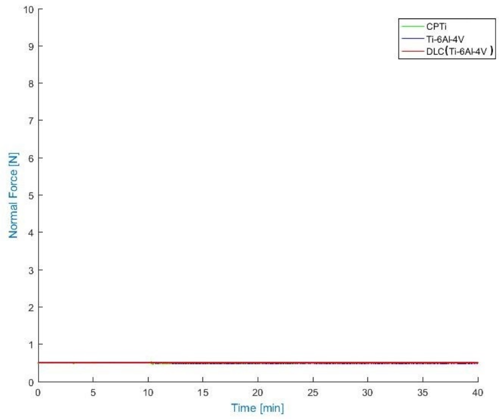


Figure 4.9: Normal Force 0.5 N protocol 1

Protocol 2

The normal force applied was 2 N and is percentage error was under the 5%, therefore making the parameter reliable (Fig. 4.10).

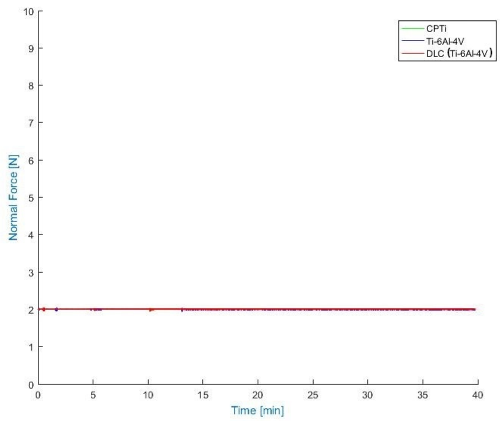


Figure 4.10: Normal Force 2 N protocol 1

4.2.2 Coefficient of friction analysis

Protocol 1

The Coefficient of Friction of the commercial titanium changes based on the stroke length(Fig. 4.11): at 0.02 mm its value is constant, changing the stroke length to 0.05 mm it rapidly changes and remains constant till the new change of stroke length, in the last passage after the change to 0.1 mm, the C.O.F. increase slowly till around 33 min when it become constant.

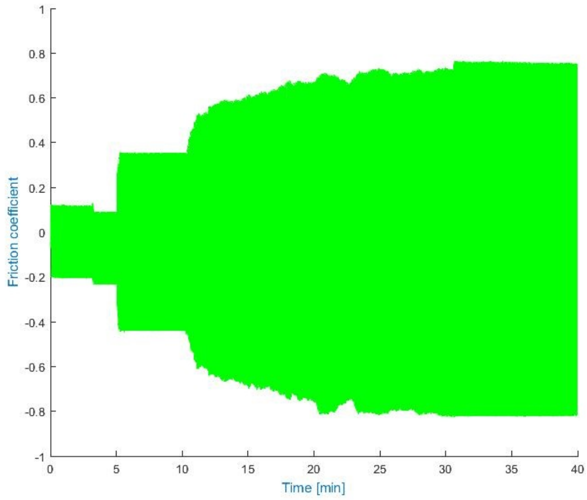


Figure 4.11: CPTi coefficient of friction protocol 1

The Coefficient of Friction of the Ti-6Al-4V changes based on the stroke length (Fig. 4.12): at 0.02 mm its value is constant, every change of the stroke length (0.02 mm to 0.05 mm and then 0.05 mm to 0.1 mm), leads to a slow increase and then to a stable value of the C.O.F.

The Coefficient of Friction of the DLC(Ti-6Al-4V) (Fig. 4.13) changes based on the stroke length: at 0.02 mm its value is constant, every change of the stroke length (0.02 mm to 0.05 mm and then 0.05 mm to 0.1 mm), leads to a small and quick increase and then to a stable value of the C.O.F.

Comparison of the coefficients of friction protocol 1 (Fig. 4.14)

It ‘s clear looking at the figure how the DLC (Ti-6Al-4V) has the better behavior in every change of stroke length. The CPTi and the Ti-6Al-4V show practically the same behavior, that increase dramatically with the increase of stroke length.

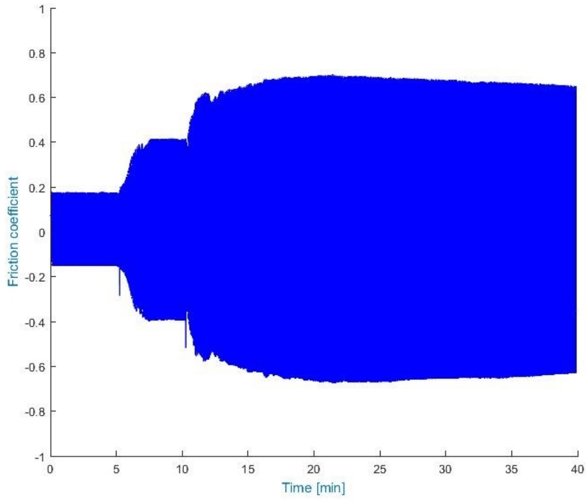


Figure 4.12: Ti-6Al-4V coefficient of friction protocol 1

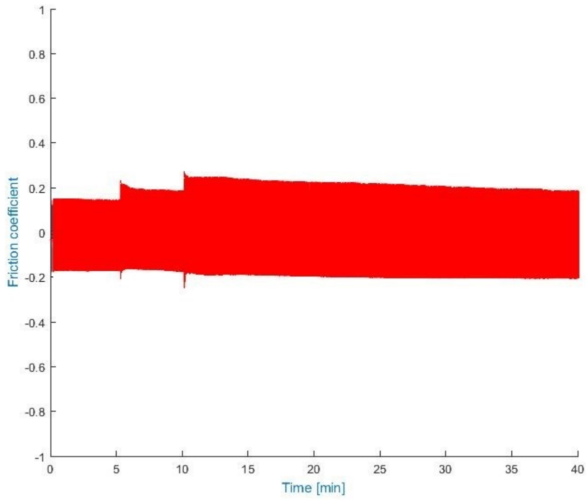


Figure 4.13: DLC(Ti-6Al-4V) coefficient of friction protocol 1

Protocol 2

The Coefficient of Friction of the commercial titanium (Fig. 4.15) changes based on the stroke length and, making a comparison with the protocol 1, its behavior is very influenced by the Normal load applied. The changes of coefficient of friction are cleanness with small increase of value, in every period of different stroke length the C.O.F remains constant

The Coefficient of Friction of the Ti-6Al-4V (Fig. 4.16) presents almost the same values and characteristics of the CPTi.

The Coefficient of Friction of the DLC (Ti-6Al-4V) (Fig. 4.17) changes as the other two previous analyzed, but the values are smaller and when there is a change of stroke length there is always a peak, a decrease, an increase, and a stabilization

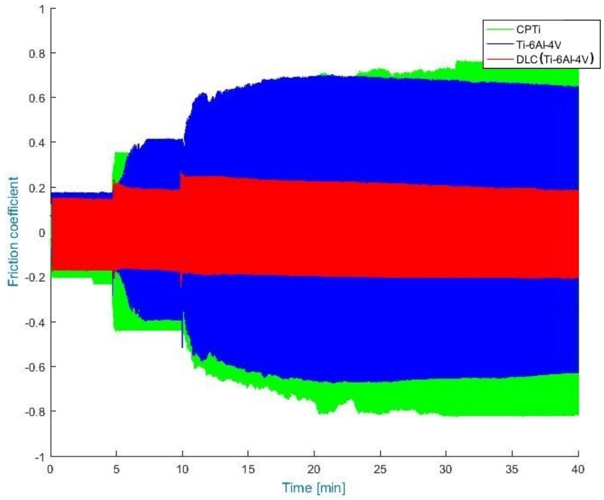


Figure 4.14: Comparison of the coefficients of friction protocol 1

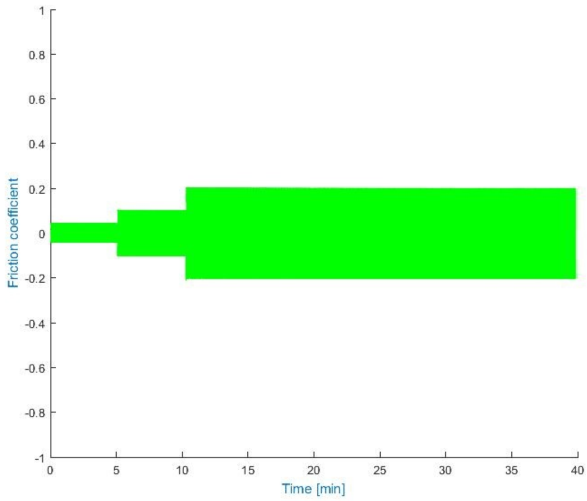


Figure 4.15: CPTi coefficient of friction protocol 2

of the C.O.F value.

Comparison of the coefficients of friction protocol 2 (Fig. 4.18)

It's clear looking at the figure how the DLC (Ti-6Al-4V) has the better behavior in every change of stroke length. However in this protocol the C.O.F values of the CPTi and the Ti-6Al-4V are nearer to the DLC (Ti-6Al-4V) one. The Titanium and its alloy have practically the same C.O.F values.

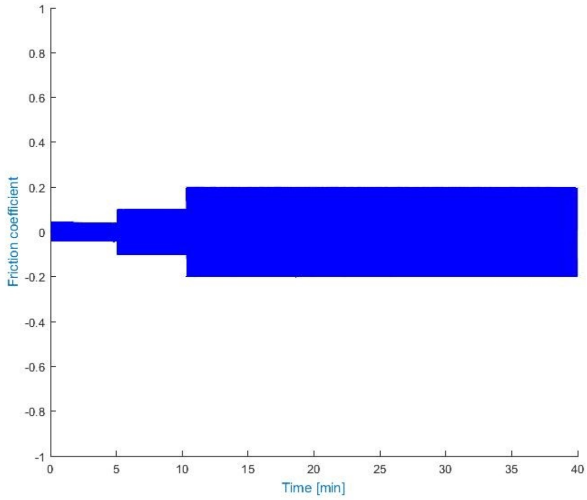


Figure 4.16: Ti-6Al-4V coefficient of friction protocol 2

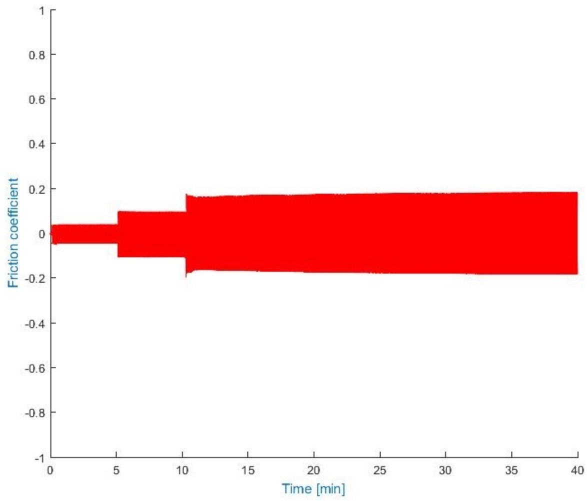


Figure 4.17: DLC(Ti-6Al-4V) coefficient of friction protocol 2

Comparison of the coefficients of friction protocol 2

The table (Fig. 4.19) shows the different behaviors, in term of friction coefficient, of the different materials and protocols. It's clear how increasing the value of the stroke length also the C.O.F will increase. The DLC (Ti-6Al-4V) has almost the same C.O.F value at 0.1 mm in both the protocols.

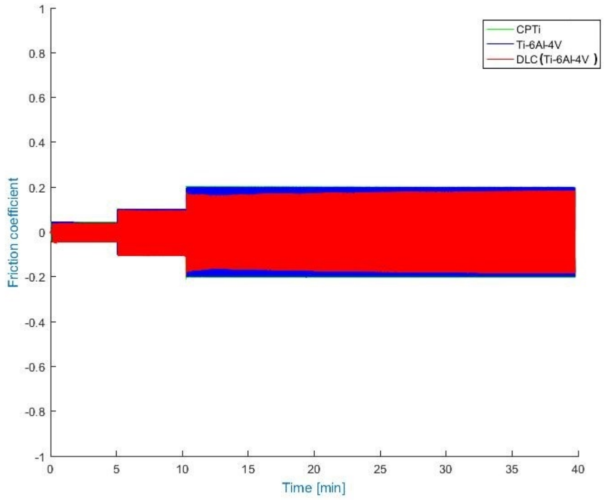


Figure 4.18: Comparison of the coefficients of friction protocol 2

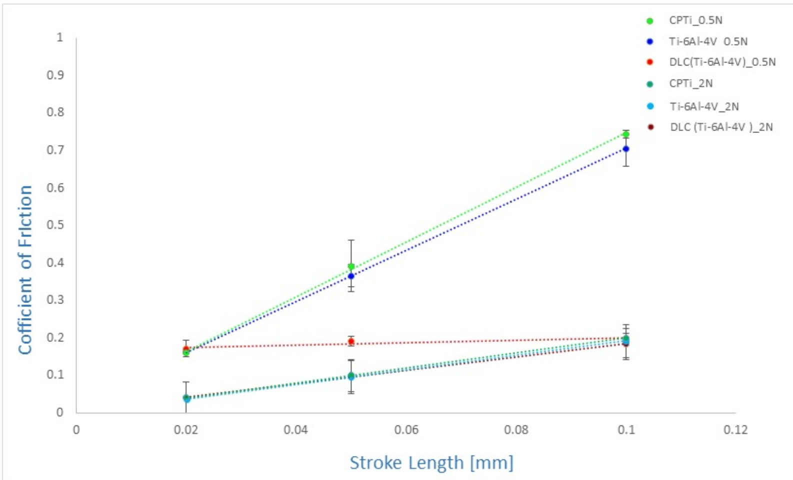


Figure 4.19: Comparison of all the coefficients of friction of both protocols

4.2.3 Morphology analysis

The morphology of the signals gives us important information, as cited before, about the condition of stick-slip, which is fundamental for reach the fretting phenomenon during the test. A pure stick is represented by a sinusoidal signal and a condition of reciprocating sliding is represented by a square wave signal[64]

In this example(Fig.4.21) there are 4 different type of stick slip:

- Regular stick-slip: the f_k peaks and the f_s peaks remains constant in values so the stick-slip is good defined.
- Irregular stick-slip: the mean of the f_s peaks is always major of the f_k peaks but the single peaks have different values irregularizing the stick-slip.
- Stiction: the force of friction increase in form f_s till reaches the f_{st} peak and then when overcome the threshold it decreases in form of f_k.

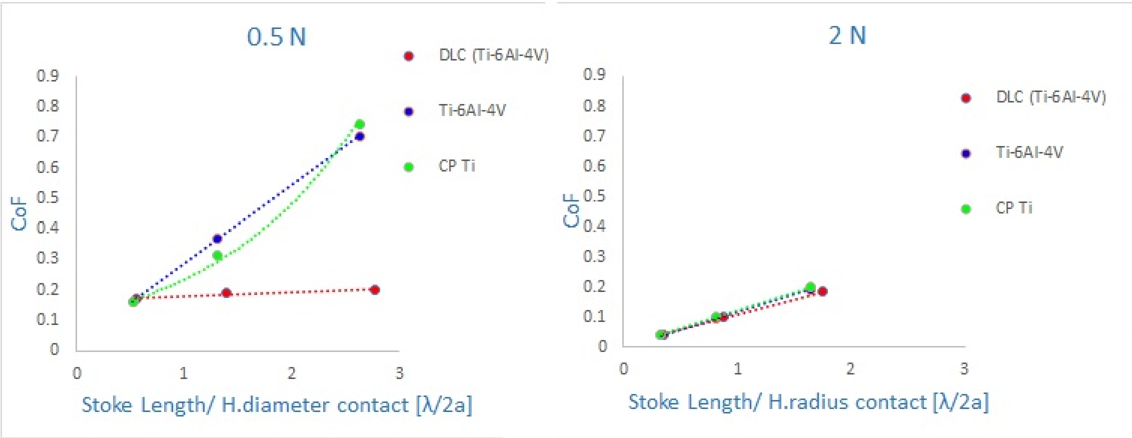


Figure 4.20: Comparison of different materials, putting in comparison coefficients of frictions vs stroke length/ hertzian contact diameter, of both the normal loads applied

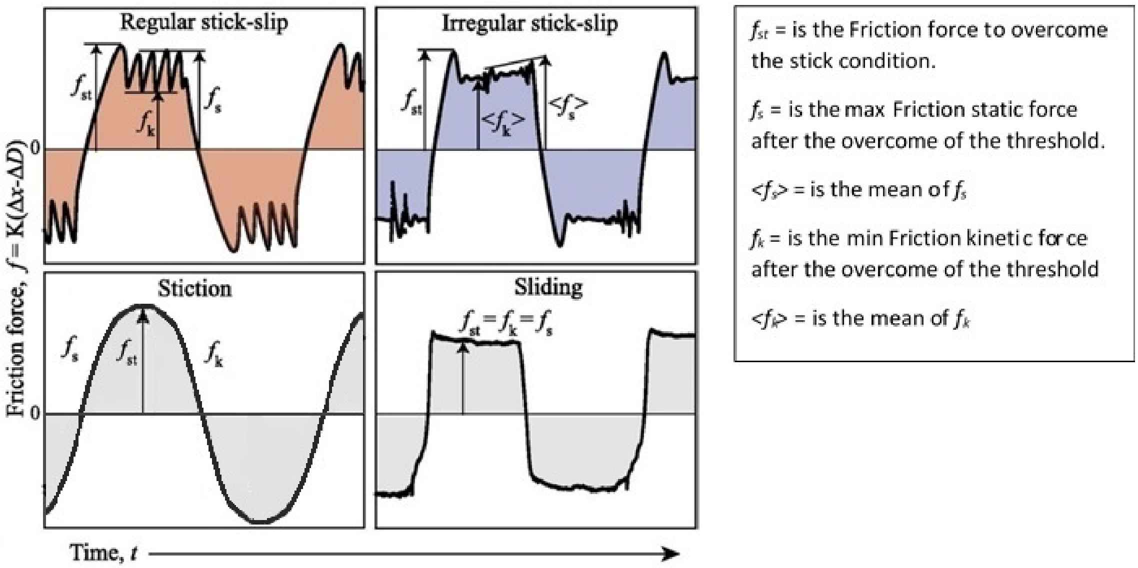


Figure 4.21: Comparison of different signal morphologies

- Sliding: f_{st}, f_s, f_k. have the same value.

The waves reported on the analysis are always of a period after fives from the start and of a period before five to the end of every window of different stroke length.

Protocol 1

Looking the morphology of the coefficient of Friction of the commercial titanium (Fig. 4.22) the waves change when the stroke length changes for then be reshaped after some minutes. At 0.02 mm the waves show a stick phenomenon. ¡When there is a change of stroke length, the signal change morphology, due to a irregular stick-slip, and after some seconds return to a stick shape.

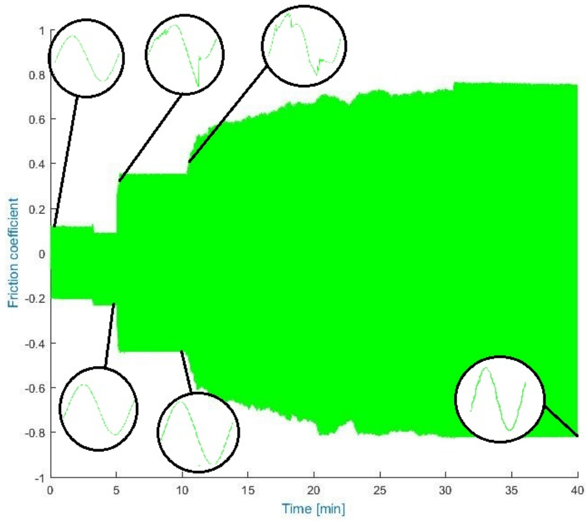


Figure 4.22: CPTi coefficient of friction signal morphology protocol 1

Looking the morphology of the coefficient of Friction of the Ti-6Al-4V (Fig. 4.23) the waves change when the stroke length changes to then be reshaped after some minutes. At 0.02 mm the waves show a stick phenomenon, while, at every change of stroke length, the signal change to a condition of irregular stick-slip, but very near to the regular one. At the end of every stroke length window the waves return to be in a stick condition shape.

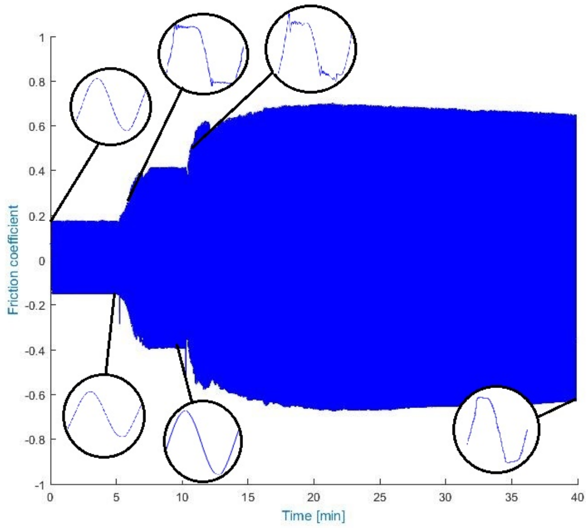


Figure 4.23: Ti-6Al-4V coefficient of friction signal morphology protocol 1

Looking the morphology of the coefficient of Friction of the DLC (Ti-6Al-4V) (Fig. 4.24) the waves change when the stroke length changes. At 0.02 mm the waves show a stick phenomenon, at 0.05 mm the waves vary in value, but the shape is generally of the irregular stick-slip. Same thing at the start of the 0.1 mm window but, finally, the waves shape begin to be near to the sliding condition.

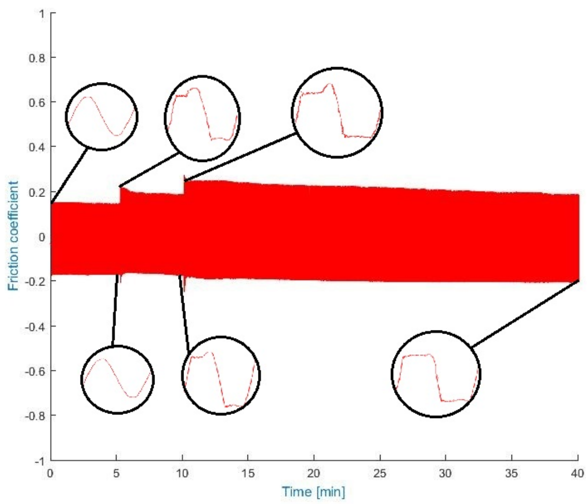


Figure 4.24: DLC (Ti-6Al-4V) coefficient of friction signal morphology protocol 1

Protocol 2

Looking the morphology of the coefficient of Friction of the commercial titanium (Fig. 4.25) it's clear, respect to the protocol 1, how the normal load applied influences the behavior of the material. The waves change only in amplitude when the stroke length changes. The waves are typical of a stick condition.

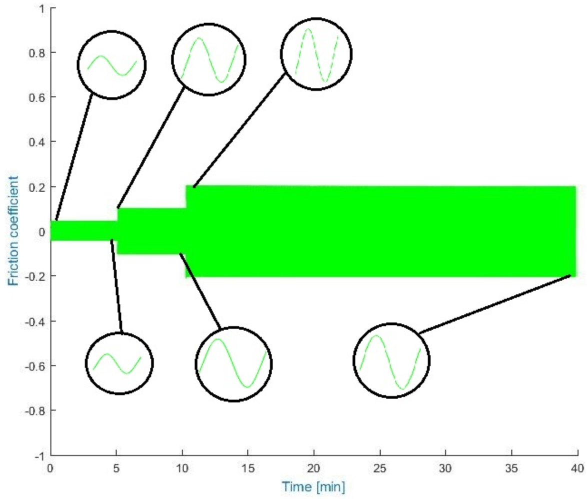


Figure 4.25: CPTi coefficient of friction signal morphology protocol 2

Looking the morphology of the coefficient of Friction of the Ti-6Al-4V titanium (Fig. 4.26) it's clear, respect to the protocol 1, how the normal load applied influences the behavior of the material. The waves change in amplitude when the stroke length changes. At 0.02 mm and 0.05 the waves have a strict flat peak, that means that the signal behavior is turning in the sliding condition.

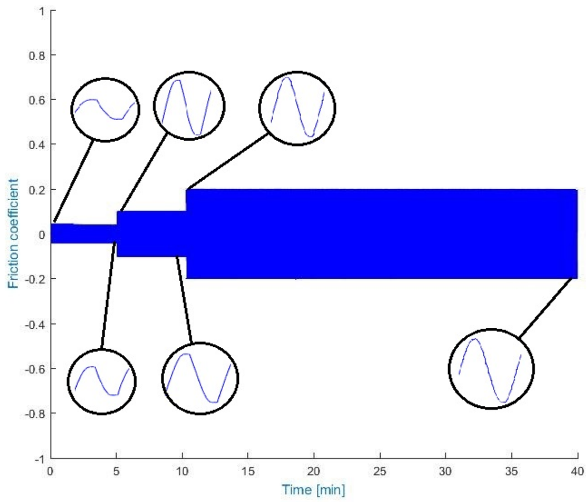


Figure 4.26: Ti-6Al-4V coefficient of friction signal morphology protocol 2

Looking the morphology of the coefficient of Friction of the DLC (Ti-6Al-4V)(Fig. 4.27) the waves change not so much in comparison to the protocol 1. At 0.05 the waves show an irregular stick slip behavior, while at 0.1 become more regular. Making a comparison with other materials it is the only one that shows a stick-slip phenomenon, but despite of it the C.O.F values are always smaller than the others.

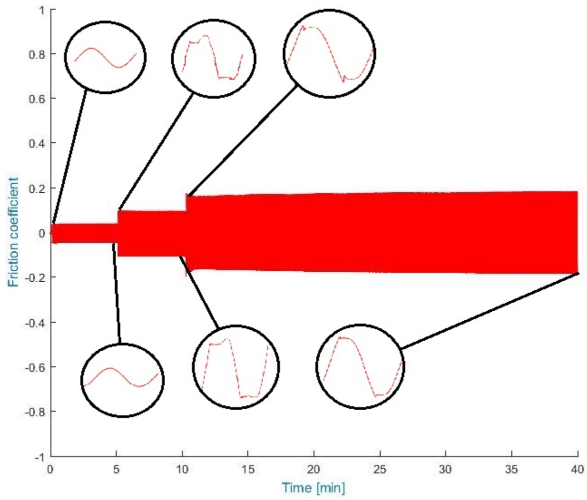


Figure 4.27: DLC (Ti-6Al-4V) coefficient of friction signal morphology protocol 2

4.2.4 fretting loops analysis

The analysis of the fretting loops is only qualitative non quantitative for two main reasons:

- The construction of the loops is obtained with post-analysis tools, that means that the representation of the loops in the test time-line is afflicted by several errors and approximations.
- Without the wear volume data the energy dissipation is unuseless for the wear characterization of the materials.

Other clarification is that the shapes obtained of the loops are extrapolated by the only relationship between C.O.F and displacement, the stiffness of the system was not used on the calculation criteria. *Protocol 1*

The protocol 1 fretting loops (Fig. 4.28) show that the DLC (Ti-6Al-4V) present a very low energy dissipation in comparison to the other two materials.

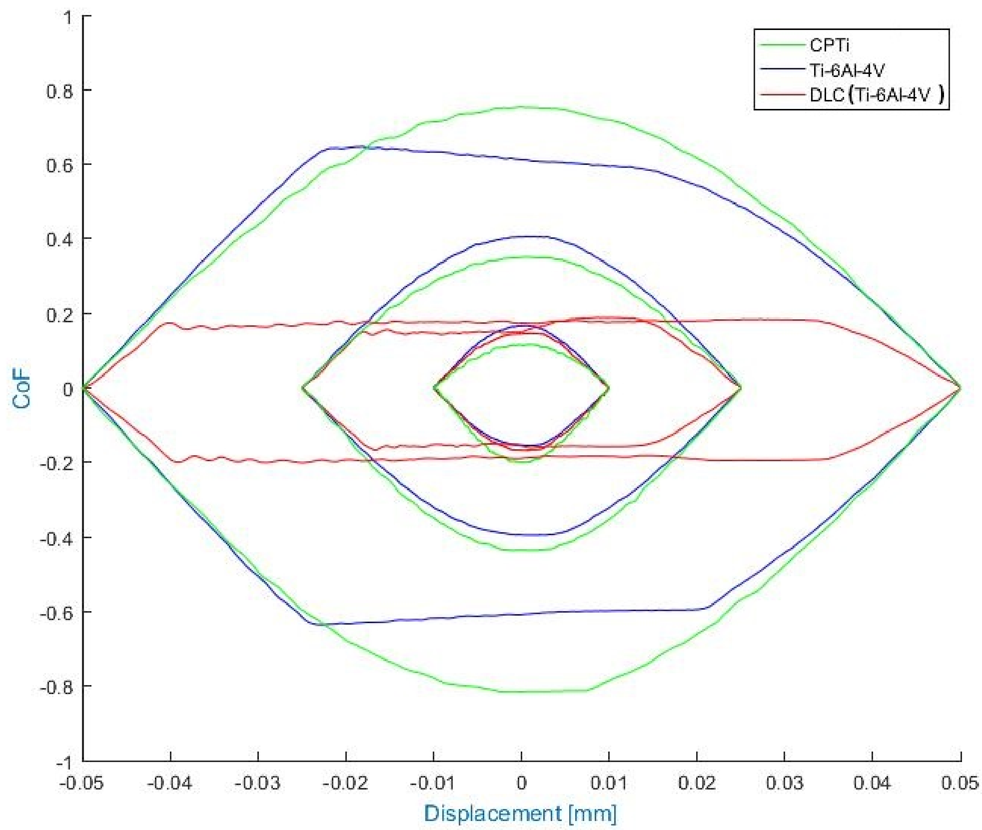


Figure 4.28: fretting loops of the different materials of the protocol 1 (5th cycle before the change from 0.02 to 0.05,5th cycle before the change from 0.05 to 0.1,5th cycle before the end of the test)

Protocol 2

The protocol 2 fretting loops (Fig. 4.29) have almost the same shape, that means that increasing the normal load the three materials dissipate the same quantity of energy.

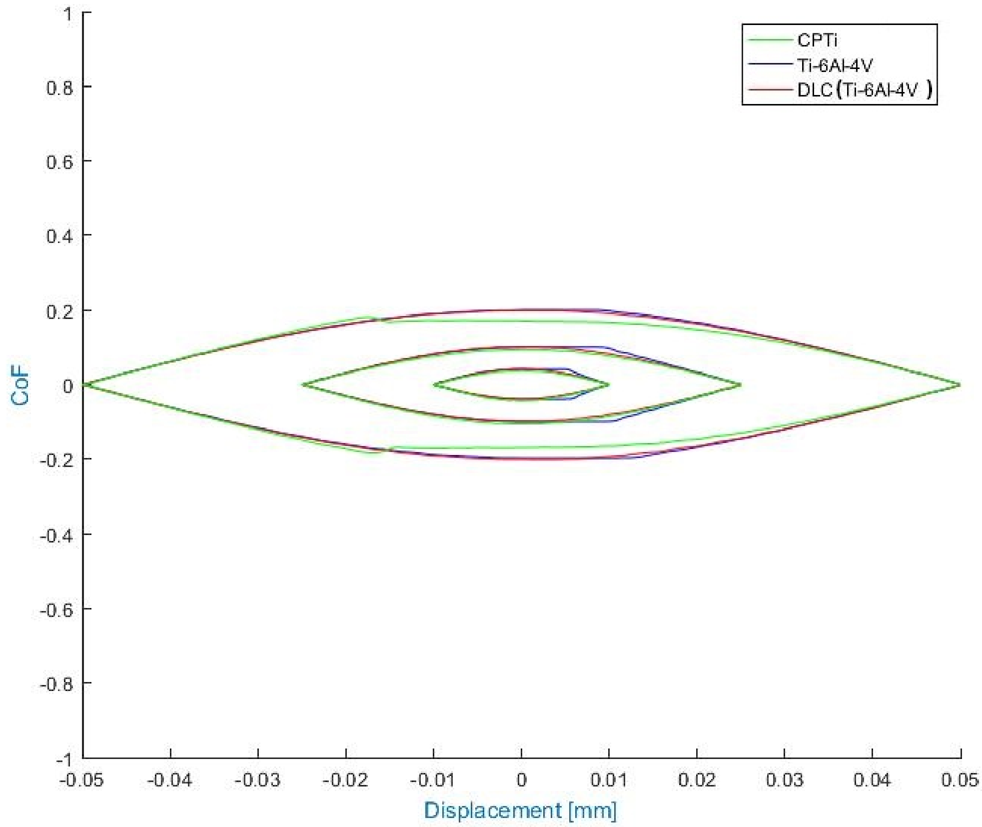


Figure 4.29: fretting loops of the different materials of the protocol 2 (5th cycle before the change from 0.02 to 0.05,5th cycle before the change from 0.05 to 0.1,5th cycle before the end of the test)

4.2.5 Wear volume

The wear volume of the micro-scars was not calculated cause the impossibility to be perceived by the instruments.

4.2.6 Wear coefficient

Without the wear volume of the materials the calculation, with both the formulas, of the wear coefficient was not performed.

Chapter 5

Conclusion

5.1 About the protocols

The protocols present some limitations that don't permit a correct analysis of all the fields of interest.

- PARAMETERS
  Looking the literature survey, the frequency and the number of cycles must be increased for have results, in terms of wear, more relevant. The first protocol probably has a right normal load, because it permits the investigation under fretting condition, but this condition is not maintained for the all duration of the test. Knowing that the other parameters should be sized in case of changes and another validation for the whole protocol must be performed. The temperature is another limit, because the protocols were performed at room temperature and not at the body one, that choice compromised the oral environment representation during the experiment.

- MATERIALS
  About the materials, the main limitation is related to the Bioxtra lubricant because it doesn't perfectly mimic the chemical composition of the oral environment. This is a big limitation, because the proteins and the enzymes have a big influence on the tribological properties of the dental implants[61].

- INSTRUMENTS
  The instrument used to evaluate the surface properties of the materials were not adapted, in relation to the entity of the scars produced by these two protocols.

- PROCEDURES
  The procedures to improve are for sure: the cleaning procedure for the surfaces after the tests, because the microscope analysis is disturbed by the Bioxtra residues, and the measure procedures, because the scars are in the scale of m and a small change in orientation of the pin can influence dramatically the measure.

5.2 About the results

The results show that the normal load has a big influence on the dissipation of the energy, especially in the Ti-6Al-4V and CPTi, that with an increase of the load applied change their behavior till reach the same C.O.F values of the DLC (Ti-6Al-4V). This change could be associated to a major stick condition of the materials, that don't permit the sliding (Necessary condition to promote the wear generation[50]).

The data suggest that at a normal load of 2N the DLC (Ti-6Al-4V) is the only one in fretting condition (but near to reciprocating sliding), so the conclusion could be that this material is not a good choice for the dental implant, but looking the data for 0.5 N we can see that the other two materials present an higher profile of fretting.

Putting these observations in relationship with the Coefficient of friction values, the DLC (Ti-6Al-4V) appears the only one stable in values and morphology and that made it the best choice to bear the masticatory cycle (the cycle presents a range of Normal load and different directions of application[20]).

5.3 Consideration of alternative solutions to the problem

About the materials in my opinion there are a lot of possible improvements. The ion release problem could be adjusted changing the fixture material with a ceramic one. The material that shall be used for all the implant components could be the Alluminazirconia, a ceramic composite that has both the good mechanical properties of Zirconia and Allumina[65]. This material has also a good tribological behaviour because, through the insertion of monoclinic Zirconia in the Allumina crystal lattice, the wear could be predicted and controlled. This composite offers also a solution to the Zirconia vs Zirconia coupling that is the worst for the wearing prevention[65].

Other type of solution is to coat the material like the DLC (Ti-6Al-4V) pins. This solution is the best with the actual manufacturing technology, because the ceramics generally are not easy to process[65]. At the moment some costume made solutions, only for the fixture, are studied for the ceramics materials, while the coating industry is already active and with good tribological results for the realization of the components.[7, 33]

About the protocols for sure the number cycles shall be raised and all the other parameter validated taking that in consideration. The work temperature shall be set to body temperature and the oral environment representation improved.

For the wear analysis the are some techniques for a better understanding of the material behavior in term of corrosion and abrasion and one of them is the anodic polarization. The procedure consists to measure the current density per unit area varying the potential applied to the pins, that are immersed in an electrolyte solution. The values obtained by this process will be directly proportional to the state of corrosion[58]. It's suggested the use of software that directly extract the fretting loops for two main reason: the data are more easy to post-process and the stiffness could be taken in consideration during the loops shaping.

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