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Review

Corrosion of Fixed Orthodontic Appliances: Causes, Concerns, and Mitigation Strategies

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Abstract: The orthodontic supply market is a prosperous billion-dollar industry, driven by an increasing demand for orthodontic appliances. The supremacy of metallic first-generation biomaterials is evident for manufacturing brackets, archwires, bands, and other components due to their well-recognized chemical inertness, spontaneous passivation, biocompatibility, and favorable mechanical properties combination. However, the oral cavity is the ultimate corrosion-promoting environment for any metallic material. In this work, the general picture of the intraoral degradation of fixed orthodontic appliances is first addressed, from the causes to the harmful effects and their oral clinical implications. Current mitigation strategies are also pointed out, including the alloys' bulk composition adjustment combined with new and advanced manufacturing processes and/or their surface treatment or coating deposition. The versatile use of thin films and coatings stands out with different deposition technologies: Many in vivo and in vitro efforts have been devoted to oral aging, from monolithic to composite architectures and micro- to nano-scale materials, to meet the best and safest oral practice demands. Unfortunately, literature data suggest that even the existing commercially available protective coatings have drawbacks and are fallible. Further multidisciplinary research is still required to effectively mitigate the corrosion behavior of fixed orthodontic appliances.

Keywords: bioalloys; biocompatibility; corrosion; intraoral aging; orthodontics; protective coatings

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1. Introduction

Orthodontics may be defined as the

> *"branch of dentistry that is concerned with the supervision, guidance and correction of the growing and mature dentofacial structures. It includes the diagnosis, prevention, interception and treatment of all forms of malocclusion of the teeth and associated alterations in their surrounding structures"*. [1]

Malocclusions—usually referred to as "*crooked*" or "*misaligned teeth*"—are a worldwide dental problem [2–5]. Technically, a malocclusion is not a disease, but rather aesthetical and/or functional misalignments between the dental arches or teeth irregularities (beyond what is considered a normal biological variation), as shown in Figure 2. Still, malocclusions can cause susceptibility to trauma and periodontal diseases [2,4,6–9]. Standard treatments for dental malocclusions involve removable or fixed orthodontic appliances.

Fixed appliances are, in general, more effective than removable ones—especially for more complex situations and/or for adult patients—and incorporate brackets, archwires, tubes, and/or bands, tightened by metallic or polymeric ligatures [10,11] (Figure 1). During treatment, a constant load is transferred from the brackets to the teeth, by using orthodontic archwires (attached to the brackets), obtaining tooth movement while adjacent bone and

tissue are remodeled [12]. Typical loads for tooth movement using fixed appliances are summarized in Table 1, usually involving values lower than 1 N.

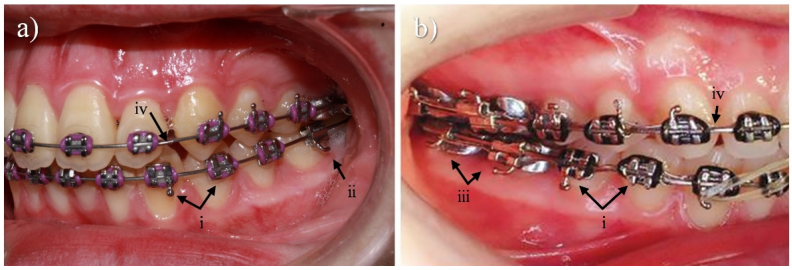

Figure 1. Typical Fixed Orthodontic Appliances: (**a**) by courtesy of Professor Sónia A. Pereira, Faculty of Medicine, University of Coimbra, Portugal; (**b**) used with permission of Dental Press Publishing from [22]. (i) Brackets with polymeric ligatures; (ii) tube; (iii) bands with attached tubes; (iv) archwires.

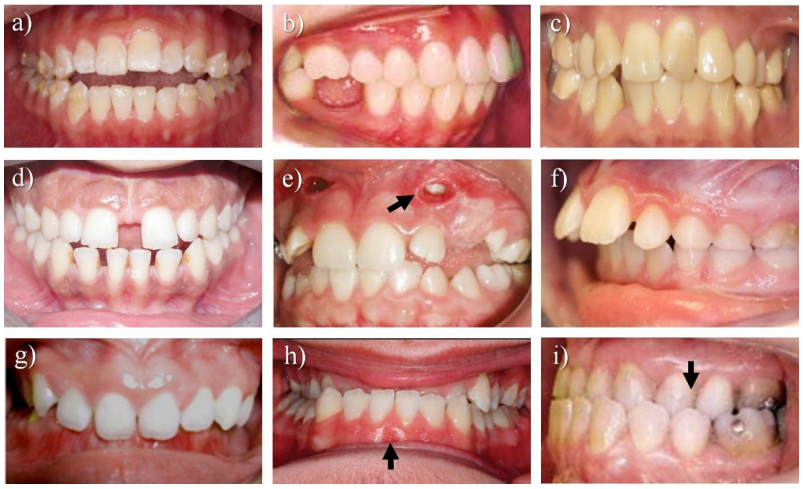

Figure 2. Some common malocclusion types: (**a**) open bite; (**b**) missing tooth; (**c**) crowding; (**d**) spacing; (**e**) impacted tooth; (**f**) overjet; (**g**) overbite; (**h**) underbite or anterior crossbite; (**i**) posterior crossbite. Adapted from [13–21]. Sources: (**a**) reproduced with permission of Oxford University Press on behalf of the European Orthodontic Society, from [13]; (**b**) reproduced with permission of Dental Press Publishing from [14]; (**c**) reproduced with permission from [15]; (**d**) reproduced with permission from [16], © Georg Thieme Verlag KG; (**e**) reproduced with permission of Dental Press Publishing from [17]; (**f**) reproduced with permission from [18]; (**g**) reprinted from [19], Copyright (2007), with permission from Elsevier; (**h**) reproduced with permission of Journal of Clinical Orthodontics from [20]; (**i**) reproduced with permission of Dental Press Publishing from [21].

A standard comprehensive orthodontic treatment may last approximately 2 years [23] and involves three sequential phases: (1st) leveling and aligning; (2nd) correction of molar relationship and space closure; and (3th) detailing and finishing [24].

In contemporary orthodontics, the market supply entails a worldwide billion-dollar industry that is expected to grow in the next few years [25]. Metallic materials are still the first choice for manufacturing fixed appliances due to their balanced set of mechanical, biological, and chemical properties [26]. Up to now, the most commonly used metallic alloys include stainless steel (SS), pure titanium (Ti) and its alloys—especially nickel–titanium (NiTi)—and cobalt–chromium (CoCr) alloys. Other metallic materials can also be found in fixed orthodontic appliances, but with a lower application range.

Table 1. Typical loads for orthodontic tooth movement (adapted with permission of Elsevier, from [27]; permission conveyed through Copyright Clearance Center, Inc.).

Movement	Description	Load (N)
Tipping	Predominant movement of the dental crown in the opposite direction.	0.34–0.59
Bodily movement (translation)	Movement that tilts the tooth until its root is in the vertical direction.	0.69–1.18
Root uprighting	Predominant movement of the root.	0.49–0.98
Rotation	Rotation of the tooth around its long axis.	0.34–0.59
Extrusion	Moving the tooth in the opposite direction to the supporting alveolar bone.	0.34–0.59
Intrusion	Moving the tooth into the supporting alveolar bone.	0.10–0.20

A clinical concern during orthodontic treatments is intraoral corrosion. Always associated with metallic ion release into the oral cavity, corrosion can be intensified by dental plaque accumulation and/or mechanical actions such as friction and fatigue stress. Several important consequences of this undesirable degradation may arise, namely enamel discoloration and demineralization, hypersensitivity, inflammatory reactions and local pain, and, in more severe cases, toxicity effects [28–32].

The need to modify the orthodontic alloys has been identified. Current research guidelines point in two main directions: (i) to adjust the alloys' bulk composition combined with new and advanced manufacturing processes; or (ii) to modify their surface, while taking advantage of the excellent mechanical properties of the bulk. The composition and microstructure of the surface can be altered by using chemical or physical methods, either by treatment or coating deposition.

The present overview is schematized in Figure 3 and partitioned into five sections. After this introductory section (Section 1), Section 2 focuses on metallic corrosion, the oral environment's aggressiveness, and its impact on orthodontic alloys. Section 3 comprises an overview of the harmful effects and clinical impact of intraoral corrosion on fixed appliances. A brief condensation concerning the mitigation strategies is presented in Section 4, describing current modifications of the orthodontic alloys. Finally, due to the utmost importance of the surface properties of any biomaterial, Section 5 provides a comprehensive overview of the protective coatings in orthodontics, from metals to ceramics and polymers, as well as their composite architectures with different reinforcement materials.

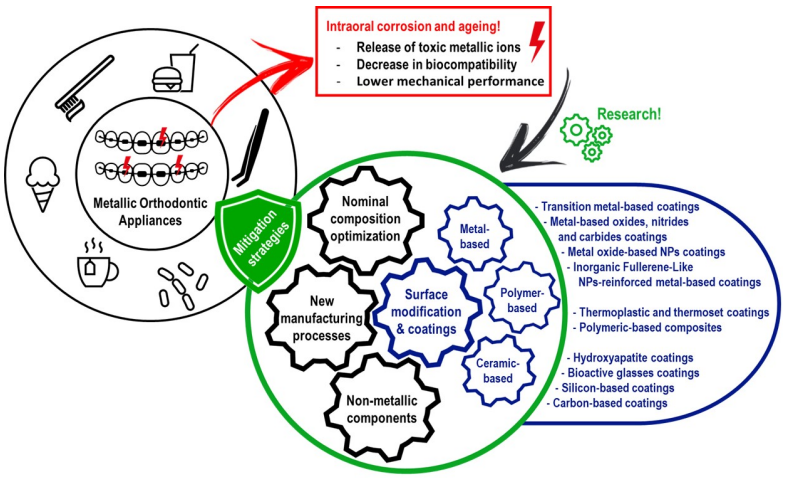

Figure 3. Schematic summary of the current review work, concerning corrosion of fixed orthodontic appliances.

2. Metallic Corrosion

This chapter focuses on the main alloys used for the manufacturing of orthodontic appliances, the characteristics of the oral environment, and their effects on the corrosion behavior of metallic alloys.

2.1. Orthodontic Alloys

Metals and alloys thrive in the medical field and are more employed as biomaterials than any other material type [26]. Today, the major metallic alloys used in orthodontic applications include stainless steel (SS), pure titanium (Ti) and its alloys—especially the nickel–titanium (NiTi)—and cobalt–chromium (CoCr) alloys. Some of the main characteristics of these bioalloys, in comparison to human molar tooth enamel, are summarized in Table 2.

Stainless steels are iron (Fe)-based alloys containing at least 12% chromium (Cr) and a maximum of 1.2% carbon (C), according to the European Standard EN 10088-1 [33] (Table 3). SS are outstanding materials for manufacturing brackets, bands, tubes, and ligatures [11,34,35], namely the austenitic 3xx series-AISI (American Iron and Steel Institute: 302, 303, 304L, and 316L), the precipitation hardening (PH) steels, as well as the duplex steels (SAF 2205) [10,36–40]. Together with Ti alloys, SS archwires are frequently used in an orthodontic treatment, especially during the 2nd and 3rd phases [41,42].

Table 2. Main characteristics of bioalloys used for manufacturing orthodontic components [37,43–46].

	Main Composition	**Young's Modulus (GPa)**	**Yield Strength (MPa)**
Human molar tooth enamel	Calcium phosphate hydroxyapatite	70–115	
Stainless steel (AISI 316L)	Fe–Cr–Ni	160–187	960–1500
Cobalt–chromium	Co–Cr–Fe–Ni	150–217	830–1200
α-Titanium	Ti (grade 4)	104	550
β-Titanium	Ti–Mo–Sn–Zr	60–68	620–690
Ti-6Al-4V	Ti–Al–V (grade 5)	100–110	830–1070
Nickel–titanium	Ni–Ti	32–36	200–500

Table 3. Composition of several orthodontic stainless-steel grades (reprinted from [10], with permission from Elsevier, after [47] (used with permission of Elsevier Science & Technology Journals; permission conveyed through Copyright Clearance Center, Inc.).

Designation	**Composition (wt.%)**									
AISI	**Fe**	**Cr**	**Ni**	**Mn**	**Mo**	**C**	**P**	**Si**	**S**	**Other**
303	Bal.	17–19	8–10	2	0.6	0.15	0.2	1.0	0.15	-
304L	Bal.	18–20	8–12	2	-	0.03	004	10	0.03	-
316L	Bal.	16–18	1014	2	2.5	0.03	0.04	1.0	0.03	-
630/17-4	Bal.	15–17	3–5	1	-	0.07	0.04	1.0	0.04	4 Cu/3 Nb
630/17-7	Bal.	16–18	6.5–7.5	1	-	0.09	0.04	1.0	0.04	0.08–1.5 Al
SAF 2205	Bal.	22	5.5	2	3	0.03	0.03	1.0	0.02	0.16 N
18–8 Plus	Bal.	8	0.16	18	1	0.15	0.045	1.0	0.03	0.5 N
431	Bal.	26	-	-	4	-	-	-	-	-
AI29	Bal.	29	0.3	0.5	4	0.02	0.035	0.35	0.01	0.5 Ti

The major advantages of these Fe–Cr alloys include their good corrosion resistance combined with their outstanding biomechanical behavior and affordable price. The key feature of the corrosion behavior is the Cr content, which is between 16 and 25 wt.% for austenitic (face-centered cubic structure, FCC) Fe–Ni–Cr alloys (Table 3). The Cr

element in the solid solution phase of SS alloys allows the development of the typical external protective chromium oxide (Cr_2O_3) thin film. Other bulk alloying elements of SS include molybdenum (Mo) and nickel (Ni > 8%), which improve the corrosion resistance effectiveness: while Ni promotes the formation of the FCC structure, Mo stabilizes the Cr-based passive layer.

However, some concerns regarding oral corrosion resistance, despite the presence of a small molybdenum (Mo) content (Table 3), and the overall biocompatibility led to the emergence of alternatives [10,11,37,42]. The high Ni nominal content in SS alloys can cause contact dermatitis (see Section 3), which has been encouraging for the development of new Ni-"free" austenitic stainless steels (see Section 4).

CoCr-based alloys have been used in orthodontics since the 1960s for manufacturing brackets and archwires [37,42,48]. With higher Cr content (>20%), these alloys surpass the SS ones in corrosion resistance—mainly in chloride environments due to the Cr-rich oxide passive layer—and biocompatibility, with higher wear resistance [42]; yet, improved ductility and resilience may be achieved (Table 2). The foremost drawbacks reported in the literature include additional heat treatments to improve mechanical performance and a more complex soldering process [10]. Currently, CoCr-based wires are commercially available in four color-coded variations according to the heat treatment applied; the blue one ("soft") is the most used due to its low yield strength compared to stainless steels [37] (Table 2).

Ti and its alloys are among the most biocompatible materials and were introduced in orthodontics in the 1980s, gaining popularity for brackets, tubes, and archwires production [11,49]. This class of metallic materials presents outstanding mechanical properties, excellent corrosion resistance (better than SS), in addition to low density (4.5 against 7.8 g/cm^3 for SS), providing a very high strength-to-weight ratio and non-eliciting allergic responses. Commercially pure titanium (α-Ti, Grade 4) and/or Ti-6Al-4V (Grade 5) brackets and β-Ti (including titanium molybdenum alloy—TMA) archwires are examples of some Ni-free components with outstanding corrosion resistance and biocompatibility [10,50]. Ti-based brackets and tubes reduce bonding failure to enamel, whereas TMA wires are ideal for certain (but not all) orthodontic situations due to the right balance of mechanical properties (e.g., low stiffness and high stringback and formability) and weldability [10,37,42]. The low elastic modulus supports the selection of β-Ti and/or NiTi alloys (Table 2) for orthodontic wires. High manufacturing cost is the most negative drawback [37].

Particular attention should be given to additional Ti-based alloys, such as NiTi and Cu–NiTi alloys, due to its high Ni nominal content. Nitinol®—which stands for "Nickel Titanium Naval Ordnance Laboratory", with near-equiatomic Ni and Ti concentrations—revolutionized orthodontics since its introduction into clinical practice in 1972 [37,51,52]. Due to its distinct mechanical properties, such as shape memory (shape memory alloy—SMA) and superelasticity behavior, this class of metals is now extensively used for the manufacture of orthodontic wires. [42,49,53]. In fact, the initial leveling stage of the orthodontic treatment (Section 1) usually involves NiTi archwires [41,42]. While the shape memory effect allows for the spontaneous recovery of the component form after being subjected to deformation higher than its elastic limit (by heating), the superelasticity tolerates a constant stress as the strain increases. After the initial elastic stress region and the stress/strain release, the NiTi alloy springs back to its original shape. Thus, high elasticity, spring back, and stored energy (Table 2) enable low-force delivery, even when malocclusions involve extreme teeth crowding. To further increase the alloys' strength and reduce energy loss, NiTi alloys have been chemically modified by copper addition (5–6% Cu)—the Cu–NiTi alloys—by acquiring a thermally activated behavior [54–56]. These wires yield lower loads on the teeth and also on deformation percentage; thus, teeth movement proceeds in a more physiological manner, preventing necrosis, hyalinization areas, and the probability of root resorption [57,58]. Other elements, such as Fe and Cr, are also added to Ni–Ti-based SMA alloys to modify their mechanical properties [59]. All Ti-based alloys spontaneously passivate by generating a titanium oxide protective film that provides good oral corrosion resistance.

Other metallic alloys can be found in fixed orthodontic appliances, but with a lower application range. The use of gold (Au)—precious metal-based alloys—for instance, was widespread before 1950 due to its higher corrosion resistance compared with alternative alloys at that time [10,60]. However, high cost and poor mechanical properties (low hardness) undermine its use, even though Au-based and Au-coated aesthetic components are still available today [10,60–64].

2.2. Intraoral Environment

The human body is an extreme environment for any metallic biomaterial [65], and the mouth is its "portal entry" [66]—an "open ecosystem" [67] in which variations in intraoral parameters are frequent and complex, leading to a unique corrosion-promoting medium.

Human saliva—99.5% water, 0.3% proteins, and 0.2% organic compounds—plays multiple important physiological functions, not only in taste, digestion, and speech but also in teeth and tissue lubrication/protection, pH buffering, and microbiological control [68–71]. The main functions of saliva and its constituents are presented in Table 4. This summary intends to reflect the saliva complexity, which is further exacerbated by other factors mentioned during this review.

Table 4. Main functions assigned to saliva and its constituents [69–72].

Function	Description	Agents
Tissue lubrication, repairing, and protection	Seromucous covering of the oral tissues. Barrier against irritants. Lubrication of hard and soft tissues, and prosthesis. Mastication, speech, and deglutition aid due to lubrication. Selective modulation of microbial adhesion to oral tissues. Modulation of dental plaque metabolism. Faster tissue repair.	Mucins and other proteins.
Clearance and pH maintenance	Acids neutralization (e.g., bicarbonate buffer). Alkalinization of dental plaque's pH through urea metabolization by its microbiome. pH modulation to prevent reaching optimal conditions for oral colonization by pathogens.	Bicarbonate, phosphate, urea, amphoteric proteins, and enzymes.
Maintenance of dental integrity	Modulation of pathogens activity to control the progression of caries and enamel damage. Maintenance of the enamel mineralization/demineralization equilibrium. The presence of fluoride in saliva enhances mineralization and forms a fluorapatite-like coating, which is more resistant to caries than the original teeth material.	Calcium, phosphate, fluoride, and several proteins (including statherin, histatins, cystatins, and proline-rich proteins).
Antibacterial activity	Selective action of protein-based immunological and non-immunological agents, allowing the growth of non-cariogenic microorganisms. Among other mechanisms, the non-immunological action involves the adhesion inhibition of colonizers to the oral tissues, namely by aggregation (clumping).	Immunoglobulins, enzymes, and other proteins (including glycoproteins, staherins, agglutinins, histidine-rich proteins, and proline-rich proteins).
Digestion, taste, and smell	Besides lubricating food and tissues, saliva starts the chemical oral digestion, namely by the initial action of the α-amylase (converting complex carbohydrates into simple sugars). The hypotonicity of saliva (low sodium, glycose, bicarbonate, and urea levels) regarding plasma, which enhances the dissolution of the substances. The presence of proteins (such as gustin) is necessary to the growth of gustatory buds.	α-amylase, gustin, lipases and other proteins.

Proteins and glycoproteins from saliva rapidly adhere to teeth enamel and any other surface placed inside the oral cavity to form a thin layer (70–100 nm), making them an important natural lubricant and oral protective film [69,73,74]. The most relevant chemical components of saliva include inorganic ions (e.g., N^+, K^+, Cl^-, F^-, $CHO_3{}^-$, $PO_4{}^{3-}$, . . .), antimicrobial factors, nitrogenous compounds, enzymes, immunoglobulins, albumin and other proteins, and glucose, among others [66,69,71,72,75]. Moreover, the chemical composition, temperature, and pH of human saliva vary between individuals and along the course of the day (circadian rhythms), also depending on the person's lifestyle, diet, and health/disease conditions [72,73,76–80].

Intraoral mean temperature usually ranges around 33–37 °C [76], but abrupt variations up to 65 °C can occur (e.g., drinking a hot coffee after eating an ice cream or drinking a glass of ice water) [77,81]. The pH of non-stimulated saliva—i.e., without consuming food or drinks—usually varies between 6 and 7, but may also oscillate from 5.3 to 7.8 [66,76,82]. A pH value below 5.5 facilitates the development of dental caries [82–84]. An acidic diet can also reduce intraoral pH to 3, for instance, due to acidic soft drinks and fruit juices (pH from 1 to 6) [68,82,85,86]. Another possible contributor to salivary pH fluctuation is regurgitated stomach acid, which has a typical pH value of 1.2 [68]—one of the intraoral problems of bulimic people and oncological patients.

The oral environment is additionally ideal for the inevitable proliferation of microorganisms. So far, over 700 bacterial species have been identified, as well as numerous fungi and viruses [66,67,73,87]. The oral microbiota co-evolved with humans in a mutualistic or even symbiotic manner: While the host provides excellent physiochemical and nutritional conditions, microorganisms (especially bacteria) play important physiological roles, including digestion, oral mucosa cell differentiation, and protection against exogenous pathogens [88,89]. Table 5 provides a summary of the main recognized oral microbiomes.

Table 5. Oral microbiome (reproduced from [73]).

Oral Bacteria Microbiome	
Saliva	*Actinobacteria*, *Bacteroides*, *Firmicutes*, *Fusobacteria*, *Proteobacteria*, *Spirochaetes*, *TM7* (The Human Microbiome Consortium)
Dental plaque	*Firmicutes*, *Actinobacteria*
Oral mucosa	*Streptococcus salivarius*, *Rothia mucilaginosa*, *Eubacterium strain FTB41*
Oral Bacteria Related to Oral Diseases	
Dental caries	*Streptococcus*, *Veillonella*, *Actinomyces*, *Granulicatella*, *Leptotrichia*, *Thiomonas*, *Bifidobacterium*, *Prevotella*, *Lactobacillus*, *Propionibacterium*, *Pseudoramibacter*, *Selenomonas*
Periapical infections (periapical periodontitis, root canal infection)	*Proteobacteria*, *Firmicutes*, *Bacteroidetes*, *Fusobacteria*, *Actinobacteria*, *Olsenella uli*, *Prevotella baroniae*, *Porphyromonas endodontalis*, *Fusobacterium nucleatum*, *Tannerella forsythia*, *Propionibacterium propionicum*, *Porphyromonas gingivalis*, *Prevotella intermedia*, *Prevotella oralis*, *Parvimonas micra*, *Porphyromonas endodontalis*, *Fusobacterium nucleatum*, *Tannerella forsythia*
Periodontal diseases (gingivitis, periodontitis)	*Actinomycetes*, *Capnocytophaga*, *Campylobacter*, *Eikenella*, *Fusobacterium*, *Prevotella*, *Porphyromonas gingivalis*, *Treponema denticola*, *Tannerella forsythia*, *Bacteroidetes* spp., *Eubacterium saphenum*, *Porphyromonas endodontalis*, *Prevotella denticola*, *Parvimonas micra*, *Peptostreptococcus* spp., *Filifactor alocis*, *Desulfobulbus* spp., *Dialister* spp., *Synergistetes*
Halitosis	*Solobacterium moorei*, *Atopobium parvulum*, *Eubacterium sulci*

Planktonic (i.e., non-attached, free-floating) bacteria are 1000 times more vulnerable to antimicrobials than when aggregated. Therefore, some species—the primary colonizers—soon physically associate with and then adhere to the glycoprotein-based film over the teeth and

biomaterials' surfaces. The mechanism of oral biofilm adhesion is described in Figure 4. Other bacterial species adhere and proliferate along with primary colonizers, forming microcolonies imbedded in an extracellular polysaccharide matrix. At this point, an oral biofilm (the dental plaque) grows: Complex groups of microcolonies positively interact with each other and even form a "primitive circulatory system" [67]. The grown (mature) oral biofilm is therefore advantageous to its inhabitants by providing nutrients to and protecting both aerobic and anaerobic colonizers—even against drugs, antimicrobial factors from saliva, and phagocytic cells [67,89,90].

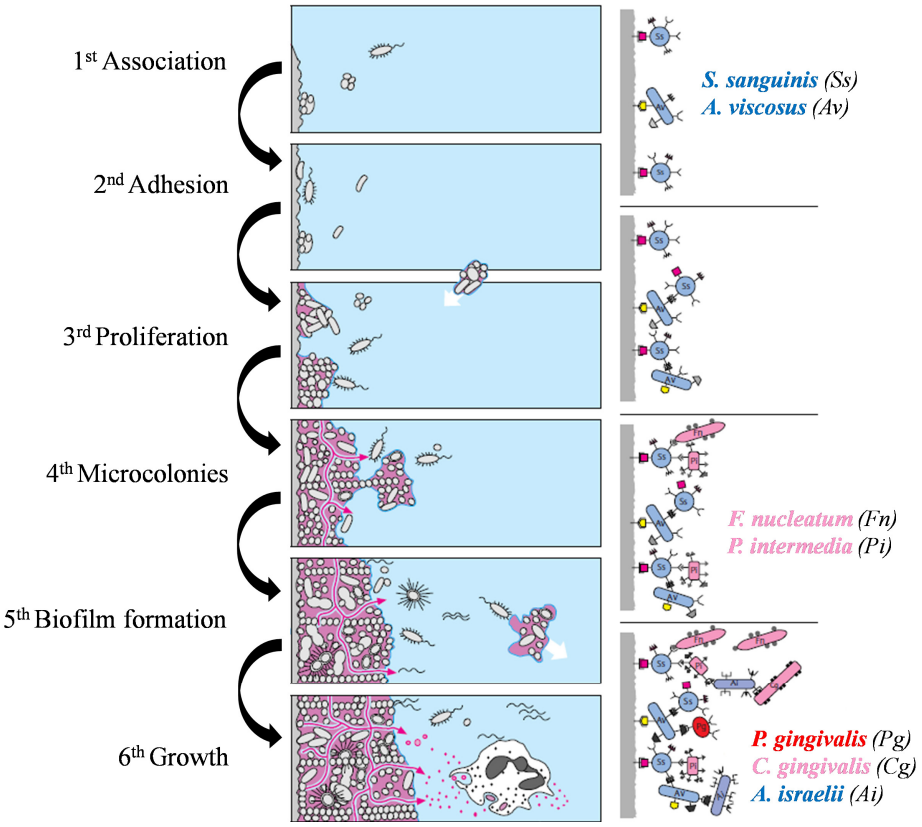

Figure 4. Six-phase oral biofilm formation mechanism over tooth and root surfaces, and representative bacterial colonizers. (used with permission of Georg Thieme Verlag KG, from [67]; permission conveyed through Copyright Clearence Center, Inc.).

While dental plaque consumes remaining food inside the mouth and protects the teeth against mechanical and chemical injuries (e.g., enamel demineralization), caries and periodontitis may occur if the host/dental plaque relationship is disturbed [73,88,89]. Some species are pathogens, and the microbiological activity of dental plaque releases several by-products into the oral cavity that can modify the chemical composition, oxygenation, and oral pH values [73,88,89]. Saliva and self-cleansing by the cheeks and tongue can naturally control biofilm growth to a certain extent. Nevertheless, oral hygiene procedures are crucial for removing dental plaque, including mechanical brushing with fluoride-containing toothpastes and mouth rinsing with fluorinated mouthwashes and elixirs [78,88,89,91,92].

In short, the intraoral environment is a highly dynamic and complex system—an ultimate degradation-promoting scenario for any biometallic material. Corrosion is a necessary (but not sufficient) condition for causing adverse biologic effects during the use of fixed orthodontic appliances.

2.3. Corrosion of Metallic Alloys

Metallic corrosion can be expressed as a "*physicochemical interaction between a metal and the environment that results in changes in the properties of the metal, and which may lead to significant impairment of the function of the metal, the environment, or the technical system, of which these form a part*" [93].

In an aqueous environment, such as the intraoral cavity, corrosion initiates through electrochemical reactions in the metal/solution interface, involving the anodic dissolution of the metal, *Me* to Me_{aq}^{2+} (oxidation, Equation (1)), and the cathodic reduction of an oxidant from the solution, Ox_{aq} to $Red\left(e_{redox}^{-}\right)_{aq}$ (reduction, Equation (2)), that is [94]:

$$Me \rightarrow Me_{aq}^{2+} + 2e_{metal}^{-} \quad (1)$$

$$2Ox_{aq} + 2e_{metal}^{-} \rightarrow 2Red\left(e_{redox}^{-}\right)_{aq} \quad (2)$$

The general charge-transfer reaction for a divalent metal can be written according to Equation (3) [94]:

$$Me + 2Ox_{aq} \rightarrow Me_{aq}^{2+} + 2Red\left(e_{redox}^{-}\right)_{aq} \quad (3)$$

Dissolved oxygen is usually the cathodic reactant—e.g., according to Equation (4)—with the production of hydroxide ions. However, other mechanisms could be more relevant under acidic conditions since protons may accept electrons produced during the anodic reaction. Typical cathodic reactions under low pH include both Equations (5) and (6), producing water or hydrogen gas, respectively [26,42,90].

$$\frac{1}{2}O_2 + H_2O + 2e^{-} \rightarrow 2OH^{-} \quad (4)$$

$$O_2 + 4H^{+} + 4e^{-} \rightarrow 2H_2O \quad (5)$$

$$2H^{+} + 2e^{-} \rightarrow H_2 \quad (6)$$

The end result of corrosion is the release of metallic ions into the electrolyte, whose extent depends on the electrolyte's nature, including its chemical composition, dissolved oxygen, and pH [42,95]. Moreover, the type of metal or alloy, its manufacturing process, and surface finishing will also influence corrosion [28,39,95–98].

Most bioalloys—mainly those containing Cr and Ti—rely on the spontaneous formation of a surface protective film. This thin oxide-based layer (some nm thick) may act as a barrier to the movement of ions—a passive film—protecting the metallic substrate against additional electrochemical corrosion [42,93,94]. Passivation is well established, being a spontaneous equilibrium of precipitation and dissolution of ions, with both active and passive films in contact with the electrolyte [99,100].

A simple passivation model [97] is presented in Figure 5. After surface hydroxylation, both metallic ions (from the alloy) and oxide ions (from the aqueous saliva) migrate through the forming film to the electrolyte and to the metal/oxide layer interface, respectively (reactions A and B, Figure 5). The electrons produced both from passivation and corrosion processes will be consumed by oxidants through redox mechanisms (reaction C, Figure 5). Therefore, electronic conduction will be essential to maintain the passivation equilibrium and the oxide film thickness. In other words, the dissolution rate must be lower than the precipitation rate [97,99]. In general, the anodic reaction of oxidation responsible for the passive film formation can be represented by Equation (7):

$$nMe + mH_2O \rightarrow Me_nO_m + 2mH^{+} + 2me^{-} \quad (7)$$

where Me_nO_m is the top metallic oxide [100].

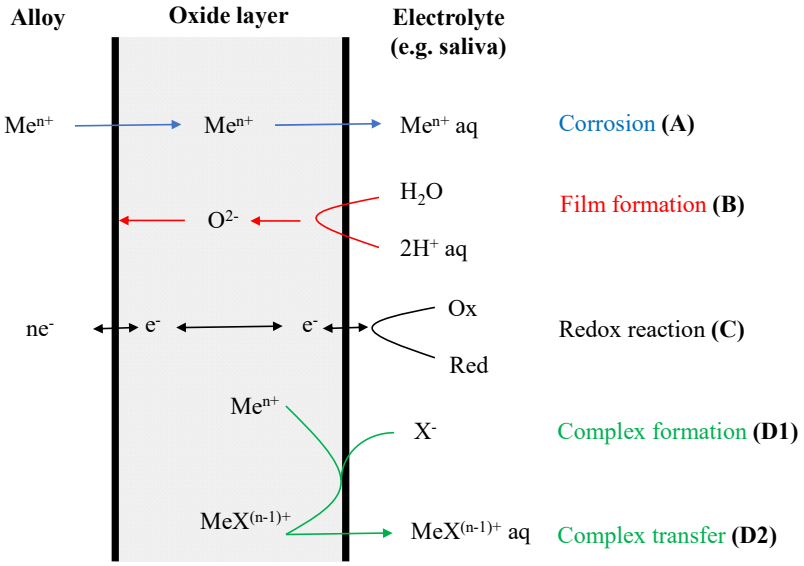

Figure 5. Passivation model of a metallic surface in an electrolyte. Me: metal element; X: complexing anions, e.g., Cl^- and F^-. (used with permission of John Wiley & Sons—Books, from [97]; permission conveyed through Copyright Clearance Center, Inc.).

It is well recognized that some elements—such as manganese (Mn), Mo, Co, and Ni—considering the normal composition of metallic alloys do not participate in passive film formation and are preferentially released into aqueous medium. If those metallic ions bond with biomolecules in vivo, they may present a certain degree of toxicity [99]. Others, however, such as Cr and Ti, are responsible for the growth of oxide films and are most likely to be less toxic [99]. The high corrosion resistance of Fe-based alloys containing Cr ($16 < Cr < 25\%$) is due to the formation of a Cr (III) oxide-hydroxide passive layer [37,101]. The insoluble Cr_2O_3 therefore prevents iron dissolution. Similarly, TiO_2-based passive layers spontaneously form on surface Ti and its alloys, being well recognized for their excellent stability [10,26,102].

Unfortunately, passive layers are fallible [95], that is, top oxide film dissolution and/or disruption may occur, driven by several environmental parameters, including fluctuations in the electrolyte chemical composition. Complexing anions can capture metallic ions from the oxide surface region (reaction D1, in Figure 5), facilitate their migration, dissolve the passive film, and release metallic ions into the saliva (reaction D2, in Figure 5) [97,103]. Aggressive ions such as chlorine (Cl^-), fluoride (F^-) [29,94,100,104], or sulphide (S^{2-}) [105], as well as mechanical damages [26,39,73,106], can disrupt the external passive oxide layer, exposing the active metallic surface for corrosion reactions to proceed. The deleterious effect of the fluoride ions—common in fluorinated prophylactic gels and mouthwash elixirs—on both SS- and Ti-based alloy passivation can be described by the following reactions, Equations (8) and (9), respectively [107].

$$Cr_2O_2 + 12F^- + 8H^+ \rightarrow 12CrHF_6^{2-} + 3H_2O \quad (8)$$

$$TiO_2 + 6F^- + 4H^+ \rightarrow TiF_6^{2-} + 2H_2O \quad (9)$$

Furthermore, both the chemical composition and the presence of defects and impurities [100] influence the thickness, stability, and composition of the external passive films [94]. Those variables, in turn, are determined by the manufacturing process and the corresponding surface finishing—mechanical/physicochemical (polishing, electro-erosion, plasma, acid/alkaline treatments, electrochemical deposition) or biochemical treatments (biological molecules to mimic biointerfaces). In the end, the balance between passive layer damage and its reconstitution will dictate the overall final corrosion resistance behavior of the biometallic alloys [94,100].

2.4. Oral Corrosion Forms

Intraoral degradation of metallic appliances is inevitable. Until today, nine basic corrosion types have been reported: uniform, galvanic, crevice, pitting, intergranular, selective leaching, stress, erosion, and microbiologically induced corrosion (MIC), which will be briefly addressed as follows.

2.4.1. Uniform Attack

Uniform corrosion is recognized as general corrosion. This degradation form seems to occur uniformly over the entire surface due to surface electrochemical reactions, almost at the same rate [81,93]. It is the most common type of oral corrosion, affecting all metallic materials at different rates [95], but sometimes it is difficult to detect—only when a significant amount of material is dissolved. All parts of a metallurgical and compositionally uniform surface should be accessible to the electrolyte (saliva) [29].

2.4.2. Galvanic Corrosion

Galvanic corrosion is observed when two different metallic surfaces—with different corrosion potentials—are joined or sufficiently close in an electrolyte solution by establishing a galvanic coupling. The more electropositive (less noble) metal or alloy becomes the anode and preferentially corrodes [26,30,81,93,95].

In orthodontics, contact between dissimilar metallic surfaces might occur in two situations: By simple contact [29,108] or through bonding processes [10,29,109]. In the first case, bracket/wire interactions are inevitable during orthodontic treatment with fixed appliances. Predictably, in certain combinations, such as in the so-common NiTi wire/SS bracket, galvanic corrosion is susceptible to occurring [29], especially in a fluoride-rich environment [108]. Recent research work [60] reported no evidence of galvanic coupling between SS lingual brackets and SS archwires, but the authors suggested caution when using fluoride-containing products during fixed orthodontic treatment with SS brackets and NiTi archwires.

Different parts of brackets or posted archwires are often made of dissimilar alloys, leading to galvanic corrosion [110]. Furthermore, brazing alloys can be used during the manufacturing of orthodontic components [10,29]. Mechanically active welding joints [95] may be reactive, increasing galvanic corrosion susceptibility accompanied by toxic metallic ion release, particularly for silver (Ag)-, copper (Cu)-, and zinc (Zn)-based welding materials [29,111,112]. A recent in vitro study [113] demonstrated that Ag ion release from Ag-soldered SS bands was an order of magnitude higher than other non-soldered SS orthodontic appliances. The authors assigned this effect to the manufacturing process used (welding).

It is generally accepted that galvanic cells can also occur in different locations of the same metallic surface due to non-uniform surface finish (e.g., roughness and chemical composition) and mechanical properties (e.g., work hardening) or even dissimilar properties of the electrolyte (pH and chemical composition) [95]. In the oral cavity, saliva is the main electrolyte, but extracellular fluids such as blood or gingival fluid are also present. Galvanic currents may take place due to the contact of the metallic surface to different biological fluids [73].

2.4.3. Crevice Corrosion

Crevice corrosion is a localized attack occurring in or near constricted places (crevices) formed by two surfaces, of which at least one is metallic [93]. This leads to a local enhancement of aggressive species and depletion of oxygen, in addition to the consequent acidification of the crevice solution due to the hydrolysis of the dissolving metal ions. Generally, metallic materials that show an affinity to pitting also suffer from crevice corrosion. The main causes include differences in metallic ions, fluoride concentration, or oxygenation between the crevice and its surroundings [28,81], associated with a pH de-

crease and a chloride ion concentration increase [10,29], which deteriorate the protective passive layer—especially on SS alloys [114].

Elastomeric or metallic ligatures are frequently used to fix orthodontic archwires to brackets, establishing ideal sites for crevice attack on brackets [109] (including on 316L SS alloy): Deep craters, fissures, and pores have been detected after long intraoral exposure, as well as extensive deterioration and perforation of the resin-fixed bracket base [28,30,81]. Daems et al. [106] also noticed this type of corrosion at regions of bracket/archwire contact or with plaque and food remnants. Other factors that reportedly cause crevice corrosion comprise the recycling process of the components [29]—not recommended in several countries—surface defects or irregularities [95,106]—including those caused by handling the components by the orthodontist during treatment (Figure 6)—and the presence of welding areas [50,115].

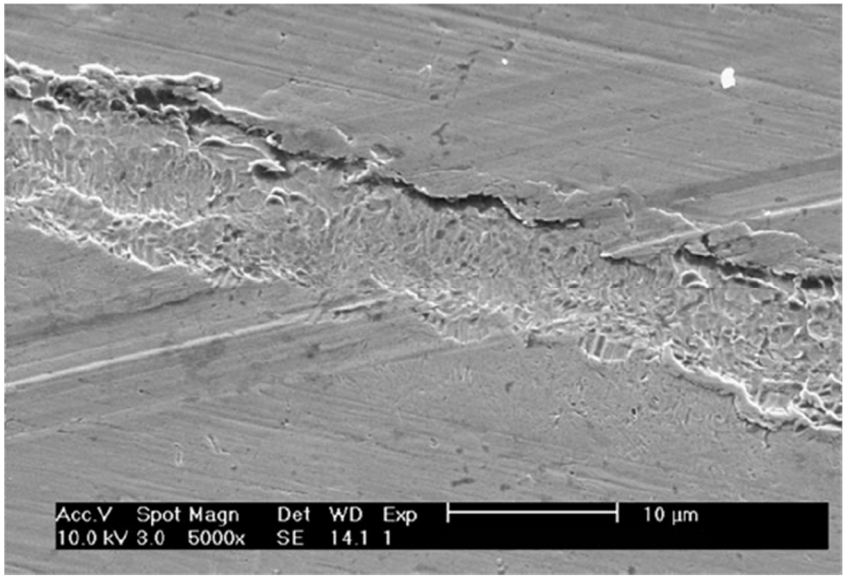

Figure 6. Crevice corrosion detected in a groove on a retrieved archwire (used for at least 6 months) caused by orthodontic pliers (reproduced by permission of Oxford University Press on behalf of the European Orthodontic Society, from [106]).

2.4.4. Pitting Corrosion

Pitting corrosion is a localized type of corrosion that initiates on metallic surfaces when the protective passive film disrupts due to mechanical and/or electrochemical attack [93,100], leading to the formation of pit holes and/or cavities. This attack has been associated with other corrosion types [114,116], such as the one caused by the well-adherent biofilm that forms during orthodontic treatments (Figure 7)—discussed further in Section 2.4.9. Aggressive ions in saliva, such as chlorine and fluoride [29,42,117]—especially under acidic conditions [101,104,118]—and food additives, such as certain spices [119,120], effectively damage surface protective oxides. Manufacturing defects on orthodontic metallic components may also increase pitting corrosion susceptibility for both SS and NiTi alloys [28–30].

The main strategy to improve the pitting resistance of SS alloys is to increase the Cr and Mo nominal content. However, the presence of non-metallic inclusions, such as manganese sulphide (MnS), is of major importance since pits usually initiate at these precipitates [121–123]. Usually, the CoCr-based alloys are resistant to pitting; the dissolution of the protective Cr_2O_3 layer into soluble ions (CrO_4^{2-}) takes place by oxidation at potentials below the oxygen evolution range [124].

2.4.5. Intergranular Corrosion

As the name suggests, intergranular corrosion occurs in microstructural planar defects along grain boundaries or in the immediate near zones, with minimal or no attack on the alloy grain itself [93,114]. The net result is an alloy fracture along these grain boundaries. SS alloys—used for manufacturing orthodontic brackets and archwires—are particularly vulnerable to this corrosion form, leading to surface staining, weakening the mechanical behavior (strength and ductility), or even failure [112,120]. Special attention should be given to heat treatment of steels [37] (or brazing/welding [95,115,125]—termed weld decay). For a prolonged period above the sensitization temperature [28,29], the formation of small precipitate particles of chromium carbide ($Cr_{23}C_6$) occurs [114]. Two major consequences arise: the SS brittleness increases and its corrosion resistance decreases, both due to the Cr-depleted zone adjacent to the grain boundary [37,95].

2.4.6. Selective Leaching

Selective leaching or dealloying is found in solid solution alloys, such as Ni–Cr-based or binary alloys containing calcium (Ca) and zinc (Zn) [26], occurring when one element is preferentially removed during the corrosion process [81,114]. This preferential release of a more reactive element from an alloy, regardless of its chemical composition [93], can occur in vivo [26,28]. Still, the effect of selective leaching seems negligible in dentistry [26,81].

2.4.7. Stress Corrosion

Stress corrosion, sometimes termed stress corrosion cracking (SCC), develops due to the influence of both applied tensile stress and a corrosive environment [28,95]. Some alloys that are virtually inert in a particular corrosive medium can become susceptible to this type of corrosion when under loads. This can seriously compromise the mechanical integrity of the material, and failure may eventually occur under low stress levels (compared with alloys in non-corrosive environments) [81,93,114]. Nitinol archwires bonded to brackets are exposed to compressive and tensile stress and might fracture during orthodontic treatment [29,126].

2.4.8. Erosion Corrosion

Erosion corrosion refers to the deterioration of a metallic material due to mechanical abrasion or wear with the combined action of the chemical attack of the corrodent fluid motion. Three subtypes are well known: Erosion, cavitation, and fretting [114]. In orthodontics, fretting corrosion is the most relevant form [81], due to the slight relative motion (vibration and slip) of two contacting metallic surfaces under load [93,114]. Surfaces of both archwires and bracket slots experience load and may undergo a cold-welding phenomenon. In addition, the required small displacements could disrupt the passive films and, consequently, increase corrosion susceptibility (e.g., by pitting) [28,29,95].

2.4.9. Microbiologically Induced Corrosion

As previously mentioned, oral microorganisms can directly or indirectly degrade metallic materials in vivo, either by metabolizing metal from the surface or by modifying the surrounding electrolyte with their metabolic by-products, respectively [95]. This form of corrosion is known as microbiologically induced corrosion (MIC) [73,114,127]. Zarasvand and Rai [90] extensively studied the MIC mechanisms, while Mystkowska et al. [73] described the intraoral process. Accordingly, oral biofilms create differential concentration cells on the metallic surfaces of three main types: oxygen concentration cells, metal concentration cells, and active–passive cells (Figure 7).

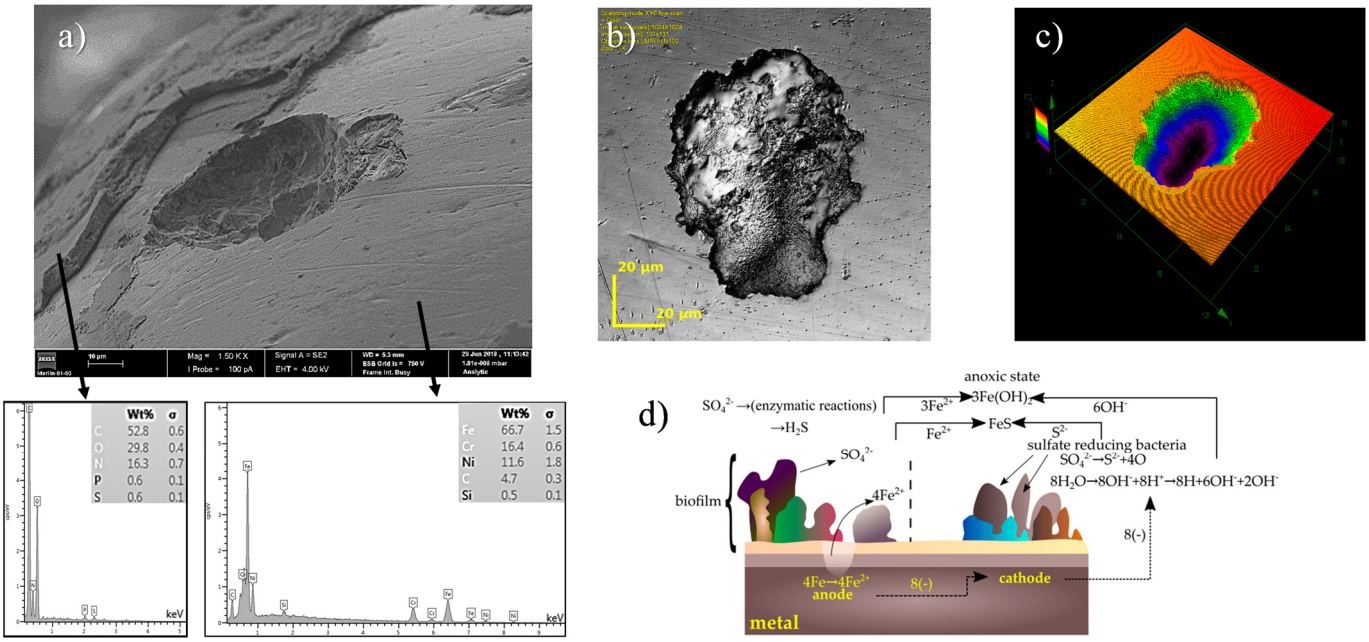

Figure 7. (**a**) SS brackets after two years of intraoral use. Scanning electron microscopy (SEM) and optical micrographs coupled to energy dispersive (EDS) chemical compositions of two distinct zones: SS alloy and pitting morphology underneath a biofilm layer. Adapted from [116]; (**b**,**c**) pitting corrosion induced by sulphate-reducing bacteria: confocal scanning microscope images after a 56-day in vitro test using *D. nigrificans* bacteria on 316LV steel; and (**d**) representation of the proposed corrosion mechanism by [128] (reproduced from [73]).

Oxygenation cells appear due to a non-uniform biofilm layer—in terms of thickness, ratio of aerobic (oxygen-consuming)/anaerobic microorganisms, or due to the presence of layers of corrosion products—that cause differences in oxygenation throughout the surface. Regions with high oxygen concentrations favor cathodic reactions (Equation (2) and Figure 8a), and the metallic surface below becomes the cathode. Conversely, in a poorly oxygenated environment, the anodic reaction is enhanced (Equation (1)), and the surface becomes the anode and corrodes. Differences in metallic ion concentration on different sites (Figure 8b) also occur due to the nature of the extracellular matrix, which has diverse composition and functional groups with different affinities to metallic ions. Under biofilm regions with low affinity to metallic ions, cathodic reactions further progress, whereas anodic dissolution of the metal increases under high-affinity biofilm sites. Finally, if a dense biofilm layer is mechanically or chemically disrupted (Figure 8c), the exposed metallic surface corrodes (the anode), while biofilm-covered regions behave as cathodes [73,90]—active–passive cells [93].

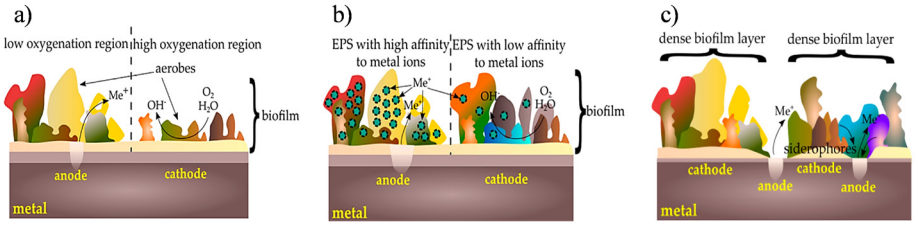

Figure 8. MIC mechanisms on a metallic surface in the oral cavity: (**a**) cells with varying degrees of oxygenation; (**b**) cells with different concentration of metal ions; and (**c**) passive–active cells. EPS: extracellular polymeric substances (reproduced from [73]).

Certain anaerobic microorganisms, such as sulfur-reducing bacteria (SRB), release corrosive metabolic products that degrade metallic alloys. The SRB can produce hydrogen gas (H_2), hydrogen sulfide (H_2S), and sulfur difluoride (F_2S, a strong local cathode), while other Gram-negative bacteria release butyric acid ($C_4H_8O_2$) and carbon dioxide (CO_2) [73,90,114].

Besides weakening and retarding the passivation mechanism of the metallic surface, H_2S is highly toxic to cells [129] and reacts with metals to form metal sulfides and atomic hydrogen. Metal sulfides may precipitate on the surface, generating new active–passive cells, while released atomic hydrogen can cause SCC [73,90,93].

Recovered SS brackets [110] and archwires [106] unequivocally revealed highly adherent biofilm deposits (Figure 9a), under which pitting or crevice corrosion occurred [106,116], as presented in Figure 7 for brackets after two years of intraoral use. Calcium-precipitating bacteria on SS orthodontic wires cause surface calcium deposits, into which chlorine ions penetrate and induce pitting [130].

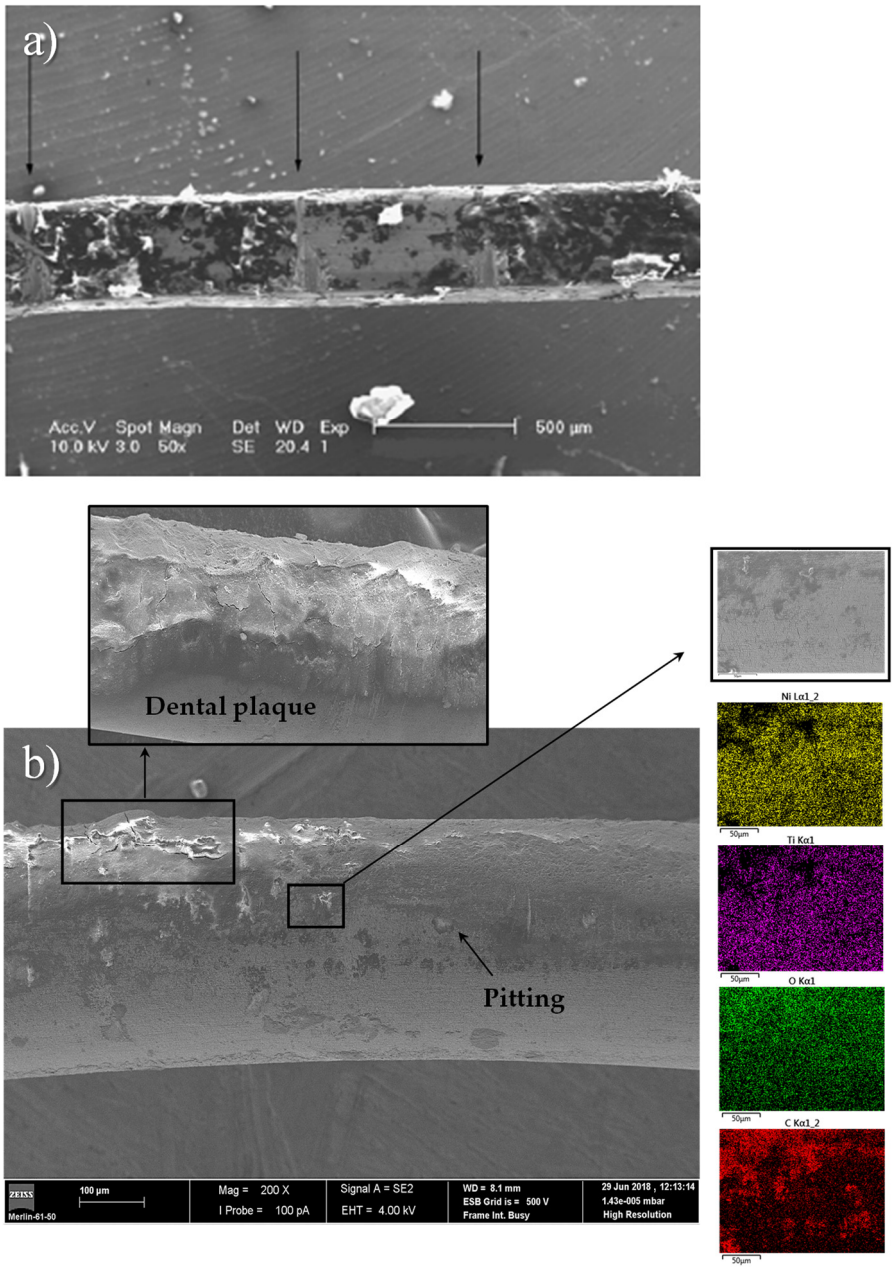

Figure 9. SEM micrographs of retrieved archwires: (**a**) SS archwires before cleaning, showing plaque and food remnants on the surface and evident marks of the location of the bracket borders (black arrows) (reproduced by permission of Oxford University Press on behalf of the European Orthodontic Society, from [106]); (**b**) SEM micrographs and EDS elemental distribution maps of an NiTi archwire after 17 weeks of intraoral use (reproduced from [116]).

Abundant biofilm layers were also observed on NiTi archwires [116] (Figure 9b), in spite of the good resistance against MIC corrosion [131,132] of the Ti-based alloys. Still, dental plaque accumulates and might enhance crevice corrosion in the welding gaps of Ti-based brackets [50,98]. Some research works [105,133] suggest that the corrosion susceptibility of Ti and its alloys decreases in the presence of bacteria, namely by inducing oxygenation cells due to bacterial metabolism.

3. Harmful Effects and Clinical Implications

The main consequences of intraoral aging of orthodontic metallic alloys are briefly presented in this chapter, namely the release of metallic ions into the oral cavity, the friction effect between components, and the consequences of using fluoride-based products during treatment with fixed orthodontic appliances.

Aging of metallic alloys is an important issue in orthodontics since both structural and morphological modifications can occur and thus negatively affect the normal clinical treatment progression. Corrosion of metallic surfaces and the presence of biofilm promote metallic ion release and roughness, which may increase friction between brackets and archwires [134–138] and extend the treatment time. Another pointed aging implication is the friction enhancement between the appliance and the mucosa, which causes oral mucosa lesions (from minor wounds to large ulcers), resulting in patients' pain and discomfort [68,139–141]. Moreover, aging decreases the resistance to fracture of metallic alloys under repeated cyclic loading [30,136]—fatigue—and could lead to premature failure of archwires [95,142] ruining the in vivo function of the biomaterial.

3.1. Release of Metallic Ions

Corrosion processes ultimately cause the release of metallic ions and particles into the oral cavity [28,99,143,144], which may interact with oral tissues and move to the gastrointestinal tract; even so, their impact on health is not yet fully understood [42,73,95,145]. Biocompatibility concerns raised among clinicians and researchers as hazardous species such as nickel, chromium, cobalt, copper, and vanadium (Ni, Cr, Co, Cu, and V, respectively) can be released from metallic appliances [26,29,144,146–148]. Back in 1975, Samitz and Katz [149]—who reviewed data related to Ni released from implanted prostheses—concluded that solubilized metal was found in tissues near implants in laboratory animals.

Multiple researchers have been trying to quantify the release of metallic ions from orthodontic appliances to assess if the concentrations can reach toxic levels for humans, both in vitro and in vivo [144,150–152]. Table 6 compiles an update of the 59 studies found in the literature regarding the in vivo measurements of metallic ions released from orthodontic appliances, sorted out by publication year.

The first study found dates back to 1991, by Gjerdet et al. [153], and measured the Ni and Fe contents in patients' saliva up to 3 months of usage. The authors found an initially higher salivary metal content that decreased over treatment time, but values were small when compared with those from dietary intake. Nonetheless, they were already alerted to the large interindividual variability found, as well as to Ni-sensitive patients [153].

Through time, Ni and Cr concentrations are almost always focused, but most studies concluded that the salivary metallic ion concentration is well below toxicity levels. In fact, dietary studies conducted in different countries obtained a daily intake of nickel between 100 and 300 μg/day from food and drinking water. Consuming Ni-enriched food (e.g., processed food) may increase this value up to 900 μg/day [95,154–160]. Haber et al. [161] estimated a toxicity reference value for Ni-sensitized populations of 4 μg Ni/kg of body weight per day, in addition to Ni in food. Concerning chromium, an average daily intake of 50–280 μg has been proposed [95,162]. However, some authors who analyzed different matrixes (oral mucosa cells, dental plaque, bone, gingiva, hair, and internal organs) found evidence of bioaccumulation that may provoke toxic effects, including DNA damage. In fact, Eliades and Athanasios [28] argued that in vivo studies measuring urinary or serum

concentrations of metallic ions in orthodontic patients may give falsely lower Ni levels due to its accumulation in an organ. Further research should therefore persist.

Among the metallic ions released into the oral cavity, Ni raises special health concerns and has been systematically studied [26,154,158,159,161,163–169], including in orthodontics [143,144,168,170–182]. The European Union (EU) currently forbids the use of Ni [183]:

1. "in any post-assemblies which are inserted into pierced ears and other pierced parts of the human body unless the rate of Nickel release from such post-assemblies is less than 0.2 $\mu g/cm^2$/week (migration limit)";
2. "in articles intended to come into direct and prolonged contact with the skin (...) if the rate of Nickel release from the parts of these articles coming into direct and prolonged contact with the skin is greater than 0.5 $\mu g/cm^2$/week";
3. "in articles referred to in point 2 where these have a non-nickel coating unless such coating is sufficient to ensure that the rate of nickel release from those parts of such articles coming into direct and prolonged contact with the skin will not exceed 0.5 $\mu g/cm^2$/week for a period of at least two years of normal use of the article" [183].

Unfortunately, biometallic alloys lie outside of this EU regulation regarding this matter. The *American Academy of Pediatrics* also expressed concerns regarding the use of Ni-containing alloys, urging the adoption of regulations similar to the EU nickel directive [168]. Dental biomaterials must still comply with several standards and regulations [184].

This transition metal (Ni) is a well-known allergen [185,186], a strong immunologic sensitizer capable of inducing delayed hypersensitive reactions [169,187], triggering cytotoxic, carcinogenic, and mutagenic effects [144,164,188,189], and affecting several cellular functions by long-term exposure to a small amount [144]. Moreover, emphasis has been given to Ni-induced genetic effects, including DNA damage and the inhibition of enzymes involved in DNA reparation [144,180]. The International Agency for Research on Cancer (IARC) classifies Ni (II) and its compounds as carcinogenic or potentially carcinogenic to humans [165].

Chromium is another well-known toxic element. Between the two most stable oxidation states, Cr(III) and Cr(VI), its hexavalent form is toxic and exhibits mutagenic, cytotoxic, and carcinogenic effects in humans [144]. Reportedly, both oxidation states were found in vitro after the corrosion of SS orthodontic brackets in artificial saliva [190].

Ni carcinogenicity, genotoxicity, and allergy are controversial in orthodontics [95,191–197]. Nonetheless, released Ni from orthodontic components can accumulate in the oral mucosa cells (see studies in Table 6) and decrease cell viability [180], while systemic toxicity should not be ignored [198]. Moreover, Kochanowska et al. [199] showed the in vivo effect of long-term exposure to metal orthodontic appliances on both the metallothionein gene expression and the induction of protein synthesis by using animal models (pigs).

Several subtle to severe intra- and/or extra-oral symptoms of allergic reactions to nickel have been reported due to the use of metallic appliances [31,147,192,200–206], as exemplified in Figure 10. Symptoms include burning sensation, stomatitis, angioedema, severe gingivitis without dental plaque, gingival hyperplasia, generalized urticaria, and widespread eczema [31,147,203,206–209]. Besides discomfort and pain for patients, orthodontists may need to replace high Ni-containing components, interrupt the treatment, and/or refer the patient to an allergologist or other specialist for further examination [201,203–205].

Ni allergy—namely extreme hypersensitive reactions—is (fortunately) rare in orthodontics [32,191,206], but may be ineffectively diagnosed: Subtle signs are easily misinterpreted as mimicking mechanical injuries or microbiologic activity [95,192,202,210]. Schuster and colleagues [192] reported allergy symptoms related to the presence of fixed appliances during treatment without intraoral signs. Corrosion products induce enamel demineralization, metallic ion incorporation, and color change [211], as well as pain and swelling of oral soft tissues, leading to secondary infections [29]. Pazzini et al. [212] concluded that patients treated with Ni-"free" (0.5–4% Ni) appliances had better gingival health and smaller blood changes when compared with those wearing conventional metallic components (13% Ni).

Another possible negative effect is the increase in antibiotic resistance of some bacteria exposed to metals and their potential transfer to medically relevant pathogens [95].

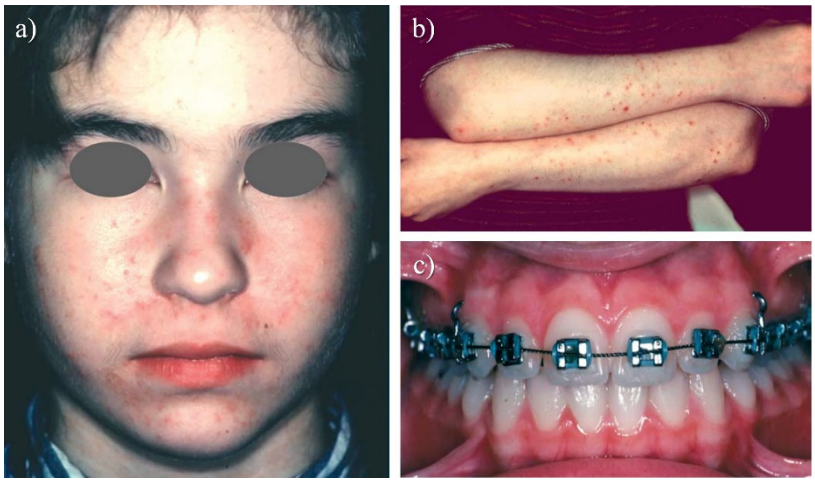

Figure 10. Examples of allergic symptoms related to the use of fixed orthodontic appliances: (**a**–**c**) extraoral allergic reaction (face and forearms) after placement of fixed appliances, without intraoral symptoms (reproduced from [192] with permission from SNCSC).

For further comprehension of the toxic effects of metallic ions released during a fixed orthodontic treatment, the reading of the outstanding review works conducted by Martín-Caméan and colleagues [144,213], and by Downarowicz and Mikulewicz [214], is recommended.

3.2. Friction in Orthodontics

Resistance to sliding is present when two surfaces come into contact with each other (e.g., bracket/wire and wire/ligature) [12,37], which is clinically relevant in orthodontics since reduced resistance to sliding can decrease treatment time [215]. Kusy and Whitley [216,217] partitioned resistance to sliding into three components: (i) Friction, "*a force that opposes every action that an orthodontist takes to move the teeth*"; (ii) binding, when the angle between the bracket slot and the archwire is high enough to promote contact between the bracket corners and the archwire; and (iii) notching, when a permanent deformation of the wire (or bracket) occurs [215–219]. Figure 11 schematizes these three contributions to resistance to sliding in a bracket/archwire contact.

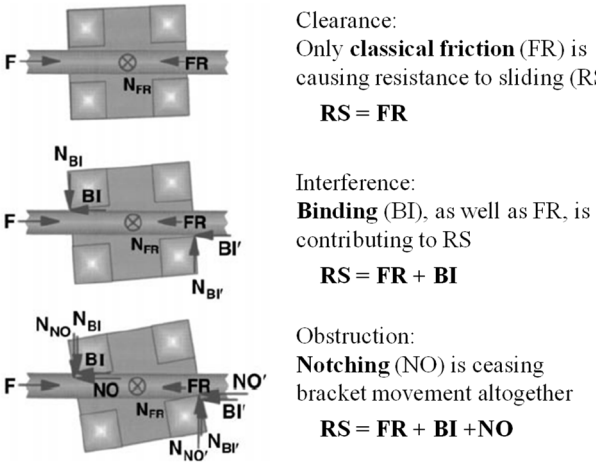

Figure 11. Schematic representations of the resistance to sliding components in a bracket/archwire contact (reproduced by permission of Oxford University Press on behalf of the European Orthodontic Society, from [218]).

Table 6. Overview of in vivo studies reporting the quantification of metallic ions release from orthodontic appliances. DL: detection limit; d.h.m.: dry hair mass; AAS: atomic absorption spectroscopy; ICP-AES/ICP-OES: inductively coupled plasma atomic/optical emission spectroscopy; ICP-MS: inductively coupled plasma mass spectroscopy; XRF: X-ray fluorescence.

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
34 patients	SS brackets and bands; NiTi archwires.	Saliva. Sampling before treatment, right away or more than 3 weeks after application, and 3–5 weeks after removal.	Ni and Fe. AAS.	Without appliances: Ni: 8.2 ppb, Fe: 148 ppb. Immediately after placement: Ni: 67.6 ppb, Fe: 488 ppb. More than 3 weeks after placement: Ni: 7.8 ppb, Fe: 172 ppb.	Significant increase in Ni and Fe concentrations and absolute masses right after placement, but not after 3 months of usage.	[153] Gjerdet et al. (1991)
31 patients	SS brackets and archwires; NiTi archwires.	Blood. Sampling before treatment, and after 3 months (with NiTi archwires) and 4–5 months (with SS archwires).	Ni. AAS.		No significant or consistent increase in Ni blood level during orthodontic treatment.	[220] Bishara et al. (1993)
47 patients	Brackets, bands and archwires.	Saliva. Sampling before and during treatment (1–2 days, 1 week, and 1 month).	Ni and Cr. AAS.	Before treatment: Ni: 68 ppb, Cr: 68 ppb. During treatment: Ni: 55–74 ppb, Cr: 69–90 ppb.	Ni and Cr concentrations were not significantly affected.	[221] Kerosuo et al. (1997)
45 patients + 15 controls	Metallic brackets, tubes, and bands; NiTi archwires.	Saliva. Sampling before and during treatment (1 week, 1 month, and 2 months).	Ni and Cr. AAS.	Before treatment: Controls: Ni: 1.16 µg/mL, Cr: 2.20 µg/mL. Patients: Ni: 0.53–0.54 µg/mL, Cr: 1.35–1.41 µg/mL. During treatment: Controls: Ni: 1.33–1.46 µg/mL, Cr: 2.50–3.43 µg/mL. Patients: Ni: 0.49–0.67 µg/mL, Cr: 0.49–1.98 µg/mL.	Fixed orthodontic appliances do not seem to significantly affect Ni and Cr concentrations in saliva.	[177] Kocadereli et al. (2000)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
100 patients	SS brackets, bands and archwires; NiTi archwires.	Saliva and Serum. Sampling before and during treatment (1 week, 1 month, 1 year and 2 years).	Ni and Cr. AAS.	Saliva: Before treatment: Ni: 4.45 ppb, Cr: 0.75 ppb. During treatment: Ni: 4.12–11.53 ppb, Cr: 0.53–1.53 ppb. Serum: Before treatment: Ni: 8.36 ppb, Cr: 6.21 ppb. During treatment: Ni: 7.87–10.27 ppb, Cr: 6.16–10.98 ppb.	The maximum levels of Ni and Cr in saliva were recorded 1 month after starting the orthodontic treatment. Below toxicity levels. The maximum levels of Ni and Cr in serum were recorded 2 months after starting the orthodontic treatment. Below toxicity levels.	[172] Ağaoğlu et al. (2001)
17 patients + 7 controls	SS brackets and archwires.	Saliva. Before and after rinsing with distilled water.	Ni, Cr and Fe. ICP-AES.	Controls before: Ni: 18 ppb, Cr: 20 ppb, Fe: 21 ppb. Controls after: Ni: 11 ppb, Cr and Fe: < DL. Patients before: Ni and Cr: < DL, Fe: 14. Patients after: Ni: 10 ppb, Cr: 27 ppb, Fe: 17 ppb.	No significant difference between controls and patients. Below toxic levels.	[222] Eliades et al. (2003)
55 patients + 30 controls	SS brackets, bands and archwires; NiTi and CoCr archwires.	Oral mucosa epithelial cells.	Ni, Co. ICP-MS.	Controls: Ni: 0.725 ppb, Co: 0.202 ppb. Patients: Ni: 2.521 ppb, Co: 0.568 ppb.	Statistically significant differences between controls and patients. DNA damage (Comet assays).	[223] Faccioni et al. (2003)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
24 patients + 24 controls	SS brackets, bands and archwires; NiTi archwires.	Saliva and dental plaque (on enamel and metallic surfaces). Sampling at approximately 16 months after starting the orthodontic treatment.	Ni. AAS.	Controls: Filtered saliva Ni: 0.004 μg/g, Saliva sediment Ni: 14.85 μg/g. Dental plaque (all tested surfaces) Ni: 0.380–0.875 μg/g. Patients: Filtered saliva Ni: 0.005 μg/g Saliva sediment Ni: 25.25 μg/g Dental plaque (all tested surfaces) Ni: 0.685–2.690 μg/g	Statistically significant Ni increase in both filter-retained fraction (saliva sediments) and dental plaque.	[178] Fors and Persson (2006)
15 patients + controls	Orthodontic appliances (not specified).	Hair. During the orthodontic treatment (not specified).	Ni. AAS.	Controls: Ni: 0.64 μg/g. Patients: Ni: 0.50 μg/g,	No statistically significant different between patients and controls.	[224] Levrini et al. (2006)
10 patients	Brackets, bands, and tubes; NiTi and SS archwires.	Hyperplastic and healthy gingiva. After 2–4 year-treatments with fixed orthodontic appliances.	Ni. AAS.	Ni in healthy gingiva: 1.81 μg/g; Ni in hyperplastic gingiva: 1.32 μg/g.	Non-significant differences between Ni content in healthy and hyperplastic gingiva, but histological differences (toxicity).	[210] Gursoy et al. (2007)
21 patients	SS brackets and bands	Urine. Before and 2 months after placement	Ni. AAS	Before placement: Ni: 17.67 μg/L. After placement: Ni: 19.89 μg/L.	Statistically significant increase in urinary Ni 2 months after placement.	[225] Menezes et al. (2007)
10 patients	SS brackets, bands, and ligatures; NiTi archwires.	Saliva. Sampling before and during treatment (1 and 3 weeks).	Ni and Cr. AAS.	Before treatment: Ni: 31.62 ppb, Cr: 38.82 ppb. After 1 week: Ni: 113.20 ppb, Cr: 83.15 ppb. After 3 weeks: Ni: 65.24 ppb, Cr: 64.21 ppb.	Statistically significant increase in salivary Ni and Cr concentrations during the orthodontic treatment when compared with the basal levels.	[181] Singh et al. (2008)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
30 patients + 30 controls	SS brackets, bands and archwires; NiTi archwires,	Saliva and mucosa cells. 1 sampling,	Ni, Cr, and Co. AAS.	Controls: Ni: 12.26 ppb, Cr: 3.46 ppb, Co: 0.44 ppb. Patients: Ni: 21.74 ppb, Cr: 4.24 ppb, Co: 0.84 ppb.	Significantly higher Ni concentrations in patients when compared with controls. No differences regarding Cr and Co levels.	[226] Amini et al. (2008)
30 patients	SS brackets	Saliva. Sampling before and after 10 min, 24 h, 7 days, 30 days, and 60 days of usage.	Ni, Cr and Fe. AAS.	Before treatment: Ni: 5.25 µg/L, Cr: 0.64 µg/L, Fe: 94.03 µg/L. During treatment: Ni: 1.69–16.01 µg/L, Cr: 0.52–1.72 µg/L, Fe: 28.31–103.58 µg/L.	Concentration peak for Ni and Cr 10 min after placing the orthodontic appliances, but no significant variations for all metals throughout the study time.	[171] De Souza and Menezes (2008)
18 patients	SS brackets and bands; NiTi archwires.	Saliva. Before and during the orthodontic treatment (immediately after placing the SS components; immediately before and after placing the NiTi archwires; and after 4 and 8 weeks).	Ni. ICP-MS.	Before placing any component: Ni: 34 µg/L. After placing SS components: Ni: 78 µg/L. After placing the archwires: Ni: 56 µg/L. During the remaining study time points: Ni: 28–34 µg/L.	Statistically significant increase in salivary Ni immediately after placing the SS and NiTi components.	[143] Petoumeno et al. (2009) and [131] Petoumeno et al. (2008)
15 patients, divided according to the bracket type	SS, Ti, and Ni-free brackets and tubes.	Oral mucosa cells. Sampling before and 30 days after placing the appliances.	Ni, Cr, Fe, Ti, Co, Mn, and Mo. ICP-MS.	Before treatment: Ni: 3.44 µg/L, Cr: 0.00 µg/L, Fe: 1.95 µg/L, Ti: 0.98 µg/L, Co: 0.00 µg/L, Mn: 0.32 µg/L, Mo: 0.13 µg/L. After 30 days: Ni: 0.00–0.04 µg/L, Cr: 0.00–0.34 µg/L, Fe: 1.24–5.36 µg/L, Ti: 0.82–3.04 µg/L, Co: 0.00 µg/L, Mn: 0.58–1.08 µg/L, Mo: 0.00 µg/L.	Increased Ti and Mn in cells exposed to the SS components. Higher Cr and Fe detected in cells exposed to Ni-free components. Increased Mn in cells exposed to Ti components. Ti components are the most biocompatible.	[227,227] Fernández-Miñano et al. (2011)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
20 patients + 20 controls	SS brackets and bands; SS and NiTi archwires.	Oral mucosa cells. Sampling after debonding (removal) of the orthodontic appliances, and 30 days later. Minimum treatment time of 18 months.	Ni and Cr. ICP-MS.	Immediately after debonding: Controls Ni: 3.86 ppb, Controls Cr: 2.71 ppb, Patients Ni: 4.09 ppb, Patients Cr: 3.63 ppb. 30 days after debonding: Controls Ni: 3.48 ppb, Controls Cr: 2.26 ppb, Patients Ni: 3.83 ppb, Patients Cr: 2.94 ppb.	No statistically significant differences between the groups regarding Ni and Cr concentrations. Genotoxic damage during orthodontic treatment reverted.	[228] Natarajan et al. (2011)
28 patients divided in 4 groups according to the brackets/archwires type combination+ 18 controls	SS brackets and bands; SS and Ti brackets; SS and NiTi archwires.	Oral mucosa cells. Sampling before and during treatment (3 and 6 months after placement).	Ni and Cr. AAS.	Before treatment: Ni: 0.52 ppb, Cr: 0.31 ppb. After 3 months: Ni: 0.68 ppb, Cr: 0.41 ppb. After 6 months: Ni: 0.78 ppb, Cr: 0.78 ppb.	Statistically significant increase in Cr content fat 3 months, as well as in Ni and Cr content after 6 months. DNA damage. SS brackets/SS archwire combination shows higher biocompatibility, whereas the Ti brackets/NiTi archwires are the less biocompatible.	[180] Hafez et al. (2011)
20 patients divided according to the bracket type	SS conventional or self-ligating brackets and bands; NiTi archwires.	Saliva. Sampling before and during treatment (1, 7 and 30 days after placement).	Ni and Cr. AAS.	Before treatment: Ni: 0.68 µg/L, Cr: 5.19–6.06 µg/L. After 1 day: Conventional—Ni: 1.95 µg/L, Cr: 21.78 µg/L. Self-ligating—Ni: 2.72 µg/L, Cr: 10.65 µg/L. After 7 days: Conventional—Ni: 2.89 µg/L, Cr: 36.69 µg/L. Self-ligating—Ni: 4.95 µg/L; Cr: 14.34 µg/L. After 30 days: Conventional—Ni: 1.18 µg/L; Cr: 8.98 µg/L. Self-ligating—Ni: 1.12 µg/L; Ce: 6.31 µg/L	Non-significant increase in Ni and Cr concentrations during the orthodontic treatment. Below dietary levels.	[170] Sahoo et al. (2011)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
30 patients + 30 controls	Hyrax appliances (which include 4 orthodontic bands with Ag-based soldering).	Saliva. Sampling before and after placement (10 min, 24 h, 7 days, 30 days, and 60 days).	Cd, Cu, Zn and Ag. AAS.	Controls: Cd: 0.15–0.18 µg/L, Cu: 3.81–6.54 µg/L, Zn: 0.01 µg/L, Ag: < DL. Patients before placement: Cd: 0.14 µg/L, Cu: 16.98 µg/L, Zn: 0.05 µg/L, Ag: 0.80 µg/L. Patients after placement: Cd: 0.18–0.71 µg/L, Cu: 12.63–70.60 µg/L, Zn: 0.05–0.20 µg/L, Ag: 2.01–11.53 µg/L.	All ions showed significant increases 10 min after placing the orthodontic appliances.	[229] Freitas et al. (2011)
28 patients + 18 controls	Orthodontic appliances (non-specified).	Hair. 1.5–2 years orthodontic treatment.	Ni, Cr, Mn, and Fe. ICP-OES.	Controls: Ni: 0.3642 µg/g d.h.m, Cr: 0.1298 µg/g d.h.m, Mn: 0.4850 µg/g d.h.m, Fe: 11.74 µg/g d.h.m. Patients: Ni: 0.5073 µg/g d.h.m, Cr: 0.1331 µg/g d.h.m, Mn: 0.5739 µg/g d.h.m, Fe: 12.22 µg/g d.h.m.	Non-significant differences in the hair metal contents between controls and patients.	[230] Mikulewicz et al. (2011c)
28 patients + 28 controls	SS brackets, and bands archwires.	Saliva. 12–18 months.	Ni and Cr. AAS.	Patients: Ni: 18.5 ng/mL, Cr: 2.6 ng/mL. Control: Ni: 11.9 ng/mL, Cr: 2.2 ng/mL.	Statistically significant difference for Ni between the two studied groups. Below toxic levels.	[231] Amini et al. (2012)
16 patients	SS brackets and tubes; NiTi archwires.	Saliva. Before the orthodontic treatment, immediately after and 8 weeks after placing the NiTi archwires.	Ni. ICP-MS.	Before treatment: Ni: 32 µg/L. Just after placing the NiTi archwire: Ni: 51 µg/L. 8 weeks after placing the NiTi archwire: Ni: 34 µg/L.	Statistically significant Ni increase just after placing the NiTi archwire. Non-significant difference after 8 weeks.	[232] Ousehal and Lazrak (2012)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
20 patients	SS brackets and bands; SS and NiTi archwires	Saliva. Sampling before and during the treatment (6 and 12 months).	Ni and Cr. AAS.	Before treatment: Ni: 9.75 µg/L, Cr: 3.86 µg/L. After 6 months: Ni: 10.37 µg/L, Cr: 4.60 µg/L. After 12 months: Ni: 8.32 µg/L, Cr: 2.04 µg/L.	Statistically insignificant change in Ni concentrations throughout the treatment time. Small but significant decrease in salivary Cr after 1 year.	[233] Amini et al. (2012)
40 patients + 50 controls	SS brackets and bands; SS and NiTi archwires.	Saliva. Sampling during the orthodontic treatment (ranging from 1 to 32 months after start, 1 sample per patients).	Ni and Cr. ICP-MS and ICP-OES.	Controls: Ni: 2.29 µg/L, Cr: 3.23 µg/L. Patients: Ni: 4.19 µg/L, Cr: 2.83 µg/L.	Statistically significant increase in salivary Ni and decrease in Cr between patients and controls. Non-toxic levels.	[234] Talic et al. (2013)
32 patients	SS brackets and bands; SS and NiTi archwires.	Saliva. Sampling before and during the treatment (20 days, and 3 and 6 months after starting).	Ni and Cr. AAS.	Before treatment: Ni: 5.76 µg/L, Cr: 2.6 µg/L. After 20 days: Ni: 6.54 µg/L, Cr: 3.68 µg/L. After 3 months: Ni: 5.13 µg/L, Cr: 3.41 µg/L. After 6 months: Ni: 5.61 µg/L, Cr: 3.39 µg/L.	Salivary Ni and Cr concentration did not significantly change.	[174] Yassaei et al. (2013)
24 patients + controls	SS brackets and NiTi archwires.	Hair. Sampling 16 weeks after starting the orthodontic treatment.	Ni. AAS.	Before treatment: Controls Ni: 0.245 µg/g, Patients Ni: 0.350 µg/g. After 16 weeks: Controls Ni: 0.382 µg/g, Patients Ni: 0.637 µg/g.	Statistically significant differences between controls and patients after 16 weeks.	[235] Abtahi et al. (2013)
30 patients	SS brackets and bands; NiTi archwires.	Saliva. Before placing the orthodontic treatment, 3 months before introducing stress; and 15 and 30 min after introducing stress.	Ni, Cr. AAS.	Before stress: Ni: 11.9–12.4 µg/L, Cr: 4.1–4.4 µg/L. After stress: Ni: 1.6–14.4 µg/L, Cr: 4.8–5.1 µg/L.	Significant increase in Ni concentrations after stress. No significant alteration in Cr concentrations.	[236] Amini et al. (2013)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
20 patients + 20 controls	SS brackets, bands, tubes, and ligatures; NiTi and SS archwires	Oral mucosa cells. Sampling between 13–15 months after starting the orthodontic treatment.	Ti, V, and Zr. ICP-MS.	Controls: Ti: 5.14 ng/g, Zr: < DL. Patients: Ti: 5.23 ng/g, Zr: 0.54 ng/g.	Non-significant differences in both groups.	[237] Martín-Cameán et al. (2014)
20 patients + 20 controls	SS brackets, bands, tubes, and ligatures; NiTi and SS archwires,	Oral mucosa cells. Sampling between 13–15 months after starting the orthodontic treatment.	Ni, Cr, Co, and Cu. ICP-MS.	Controls: Ni: 4.3 µg/L, Cr: 2.3 µg/L, Co: 0.6 µg/L, Cu: 4.9 µg/L. Patients: Ni: 24.8 µg/L, Cr: 17.5 µg/L, Co: 11.6 µg/L, Cu: 8.5 µg/L.	Significantly higher values for all metals for patients when compared with controls.	[238] Martín-Cameán et al. (2014)
70 patients + 56 controls	SS brackets, bands, tubes, and ligatures; NiTi and SS archwires.	Hair. Minimum duration of the orthodontic treatment of 24 months.	Ni, Cr, Fe, Mn, Cu. AAS.	Controls: Ni: 0.36 µg/g, Cr: 0.36 µg/g, Fe: 25.3 µg/g, Cu: 33 µg/g, Mn: 0.23 µg/g. Patients: Ni: 0.33 µg/g, Cr: 0.33 µg/g, Fe: 24.86 µg/g, Cu: 24 µg/g, Mn: 0.42 µg/g.	Significant increase for Mn concentrations only.	[239] Martín-Cameán et al. (2014)
24 pigs (12 controls)	SS plates simulating orthodontic appliances.	Hair, kidneys, liver, lungs, aorta, and oral mucosa. Up to 6 months.	Ni, Cr, Fe, Cd, Co, Cu, Mn, Mo, Si and Zn. ICP-OES.	Higher increase in toxic metals registered in the aorta (for Ni), cheek (for Ni) and hair (for Cr).	Products of corrosion passed into selected tissues of pigs. Below toxicity levels.	[240] Mikulewicz et al. (2014)
47 patients	SS brackets and ligatures; NiTi archwires.	Hair. Sampling before and during the orthodontic treatment (4, 8 and 12 months).	Ni, Cr and Fe. ICP-OES.	Before treatment: Ni: 0.275 mg/kg Cr: 0.0201 mg/kg Fe: 13.2 mg/kg After 1 year: Ni: 0.422 mg/kg Cr: 0.158 mg/kg Fe: 14.2 mg/kg	Statistically significant increase in Cr content only. Below toxicity levels.	[241] Mikulewicz et al. (2015)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
30 patients, divided in two groups according to the type of brackets	Conventional or MIM SS brackets and tubes; NiTi archwires.	Saliva. Immediately before and 60 days after starting the treatment.	Ni and Cr. AAS.	Before the treatment: Conventional—Ni: 7.12 µg/L, Cr: 0.25 µg/L. MIM—Ni: 8.62 µg/L, Cr: 0.42 µg/L. 60 days after treatment: Conventional—Ni: 12.57 µg/L; Cr: 0.35 µg/L. MIM—Ni: 8.86 µg/L, Cr: 0.26 µg/L.	Significant increase in Ni content in each group. Not significant differences between the groups.	[242] Amini et al. (2015)
24 patients	SS brackets and bands; NiTi and SS archwires.	Hair. Immediately before and 6 months after starting the treatment.	Ni and Cr. AAS.	Before the treatment: Ni: 0.1380 µg/g d.h.m, Cr: 0.1455 µg/g d.h.m. After 6 months: Ni: 0.6715 µg/g d.h.m, Cr: 0.1683 µg/g d.h.m.	Ni and Cr content in hair significantly increased (387 and 16%, respectively).	[243] Amini et al. (2015)
13 patients	SS brackets, bands, tubes, lingual sheath, transpalatal arch and archwires; NiTi archwires.	Saliva. Sampling before and during the orthodontic treatment (1 week, and 1 and 3 months after placement).	Ni and Cr. AAS.	Before treatment: Ni: 1.156 µg/L, Cr: 11.570 µg/L. After 1 week: Ni: 6.841 µg/L, Cr: 70.386 µg/L. After 1 month: Ni: 3.403 µg/L, Cr: 21.254 µg/L. After 3 months: Ni: 3.124 µg/L, Cr: 20.002 µg/L.	Significant increase in salivary Ni and Cr after starting the orthodontic treatment. Peak concentrations 1 week after placement.	[176] Dwivedi et al. (2015)
30 patients	SS brackets and bands; NiTi and SS archwires.	Saliva. Sampling before and during treatment (after aligning phase and 10–12 months after placement).	Ni and Cr. ICP-MS.	Before treatment: Ni: 48.78 ppb, Cr: 69.74 ppb. After aligning stage: Ni: 59.19 ppb, Cr: 102.68 ppb. 10–12 months after start: Ni: 46.33 ppb, Cr: 87.07 ppb.	Significant increase in salivary Ni and Cr after the initial aligning phase.	[182] Nayak et al. (2015)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
50 patients with 4–6 months of fixed orthodontic treatment	Fixed orthodontic appliances (not specified).	Saliva. Sampling after 1 week without using mobile phone (controls); then after 1 week of regular usage (experimental group).	Ni. ICP-MS.	Experimental group: Ni: 16.22 ng/L. Controls: Ni: 12.84 ng/L.	Statistically significant increase in Ni release for the experimental group when compared with the controls.	[244] Saghiri et al. (2015)
1 patient	Fixed appliances (not specified).	Saliva, alveolar bone, and gingiva. Collection during periodontal cosmetic surgery and exostosis removal.	Ni. AAS.	Ni in saliva: 986.4 ppb. Ni in bone: 779.5 ppb. Ni in gingiva: 620.5 ppb.	High Ni accumulation in each sample type.	[245] Arcila et al. (2015)
30 patients	SS bands and closed coil springs; SS self-ligating brackets with NiTi clip; NiTi archwire and open springs.	Saliva. Sampling before and during the orthodontic treatment (immediately after placing brackets and bands and 2 weeks later; immediately after placing the archwires and 4 and 8 weeks later).	Ni. ICP-MS.	Before treatment: Ni: 21.85 μg/L. Immediately after placing brackets and bands: Ni: 85.34 μg/L. Immediately after placing the NiTi archwires: Ni: 57.74 μg/L. For the remaining sampling times: Ni: 13.73–19.83 μg/L.	Significant increase in salivary Ni concentrations after brackets and bands insertion, as well as after placing the archwire. Return to basal levels after 4 weeks. Below dietary intake.	[246] Gölz et al. (2016)
24 patients	SS brackets; NiTi archwires.	Gingival crevicular fluid. Sampling before and during treatment (1 and 6 months).	Ni and Cr. AAS.	Before treatment: Ni: 3.894 μg/g, Cr: 1.978 μg/g. 1 month after starting: Ni: 5.913 μg/g, Cr: 4.135 μg/g. 6 months after starting: Ni: 19.810 μg/g, Cr: 13.760 μg/g.	Significant increase in Ni and Cr (up to 510 and 700%, respectively) during the treatment, as well as gingival inflammation promotion.	[247] Amini et al. (2016)
42 patients, divided according to the bracket type	MIM tubes and SS brackets; NiTi, Cu-NiTi, or epoxy-coated NiTi archwire.	Saliva. Sampling before and 2 months after starting the orthodontic treatment.	Ni. AAS.	Before treatment: Ni: 10.4571 μg/L. After 2 months: Ni: 11.0799 μg/L.	Statistically significant increase in salivary Ni concentration but depends on the archwire type.	[173] Masjedi et al. (2016)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
10 patients	SS brackets and ligatures; bands with tubes and lingual sheaths (not specified); NiTi archwires.	Saliva. Sampling before and during treatment (10 days and 1 month after placement).	Ni and Cr. ICP-OES.	Before treatment: Ni: 0.0039 mg/L, Cr: 0.0024 mg/L. After 10 days: Ni: 0.0288 mg/L, Cr: 0.0037 mg/L. After 30 days: Ni: 0.0370 mg/L, Cr: 0.0103 mg/L.	Statistically significant increase in salivary Ni after 10 and 30 days when compared with controls, as well as for salivary Cr between the 10th and the 30th day.	[248] Kumar et al. (2016)
47 patients with different dietary habits (coffee, yoghurt, juice and vinegar consumption)	Fixed orthodontic appliances (not specified).	Human hair. Sampling at the beginning of the treatment, and after 4, 8 and 12 months.	Ni and Cr. ICP-OES.	At the beginning: Ni: 0.131–0.331 mg/kg, Cr: 0.00578–0.0338 mg/kg. After 4 months: Ni: 0.222–0.505 mg/kg, Cr: 0.0620–0.446 mg/kg. After 8 months: Ni: 0.252–0.444 mg/kg, Cr: 0.0862–0.292 mg/kg. After 12 months: Ni: 0.207–0.500 mg/kg, Cr: 0.124–0.191 mg/kg.	Consuming foods and drinks with low pH can intensify metal release of Cr and Ni during the orthodontic treatment.	[249] Wołowiec et al. (2017)
30 patients	Fixed orthodontic appliances (not specified).	Saliva, biofilm, and oral mucosa cells. Before and during orthodontic treatment (1 week and 6 months).	Ni. AAS.	Before treatment: Saliva—Ni: 2.213 ppm, Biofilm—Ni: 4.943 ppm, Oral mucosa—Ni: 3.327 ppm. After 1 week: Saliva—Ni: 2.627 ppm, Biofilm—Ni: 5.75 ppm, Oral mucosa—Ni: 3.683 ppm. After 6 months: Saliva—Ni: 3.03 ppm, Biofilm—Ni: 6.917 ppm, Oral mucosa—Ni: 3.143 ppm.	Significant increase in Ni levels, especially in biofilm samples.	[250] Causado-Vitola et al. (2017)
46 patients, divided according to the bracket type	SS MIM or conventional brackets and tubes; NiTi and SS archwires.	Hair. Sampling before and 6 months after starting the orthodontic treatment.	Ni and Cr. AAS.	Before treatment: Ni: 0.1600 µg/g d.h.m, Cr: 0.1657 µg/g d.h.m. After 6 months: Ni: 0.3199 µg/g d.h.m, Cr: 0.3066 µg/g d.h.m.	Statistically significant Ni and Cr content increase, regardless the bracket type.	[251] Masjedi et al. (2017)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
37 patients	Metallic Fixed appliances (not specified).	Saliva. Sampling immediately before placing the fixed appliances, and after 1 and 24 weeks.	Ni. AAS.		Increased salivary Ni concentration, probably responsible for modifying the oxidative/antioxidative balance of saliva.	[252] Buckzo et al. (2017)
60 patients + 30 controls	Conventional appliances: SS brackets and bands, and NiTi archwires; aesthetic appliances: polycarbonate brackets and tubes and Rh-coated NiTi archwires.	Saliva. Sampling from patients undergoing an orthodontic treatment for 1–6 months. One collection per patient.	Ni, Cr, Fe, and Cu. Total reflection XRF.	Controls: Ni: 4.14 µg/L, Cr: 10.32 µg/L, Fe: 32.04, Cu: 11.40. Patients with conventional appliances: Ni: 22.20 µg/L, Cr: 89.45 µg/L, Fe: 517.77 µg/L, Cu: 15.10 µg/L.	No significant differences regarding Ni and Cr concentrations between conventional or aesthetical and control groups. Ni and Cr influenced by the type of appliances. No differences in Fe and Cu between groups.	[253,253] Lages et al. (2017)
42 patients, divided in two groups (mobile phone users and nun-users)	Fixed orthodontic appliances (not specified).	Saliva. 6–9 months after placement.	Ni. ICP-OES.	Mobile phone users: Ni: 0.012 ppb. Non-users: Ni: 0.0083 ppb.	Mobile phone radiations can influence Ni ion release, but a statistically non-significant difference was obtained.	[254] Nanjannawar et al. (2017)
30 patients	SS brackets and NiTi archwire	Gingival crevicular fluid. Sampling before and during treatment (1 and 6 month)	Ni and Cr. AAS	Before treatment: Ni: 3.2 µg/g Cr: 4.1 µg/g 1 month after starting: Ni: 4.5 µg/g Cr: 4.9 µg/g 6 months after starting: Ni: 14.2 µg/g Cr: 21.4 µg/g	Significant increase in Ni and Cr concentrations during the orthodontic treatment.	[255] Bhasin et al. (2017)
24 patients + 28 controls		Scalp hair. Sampling before and 1 year after placement.	Ni and Cr. AAS.	Before: Controls—Ni: 0.085 µg/g, Cr: 0.299 µg/g. Patients—Ni: 0.061 µg/g, Cr: 0.304 µg/g. 1 year after: Controls—Ni: 0.086 µg/g, Cr: 0.258 µg/g. Patients—Ni: 0.149 µg/g, Cr: 0.339 µg/g.	Statistically significant differences in both Ni and Cr contents in scalp hair between the groups after 1 year of orthodontic treatment.	[256] Jamshidi et al. (2018)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
42 patients with metallic brackets + 42 with ceramic brackets	SS or ceramic brackets; NiTi archwires.	Saliva. Sampling before and 6 months after starting the orthodontic treatment.	Ni, Cr, Ti, Co, Cu, and Zn. ICP-MS.	Metallic brackets: Before: Ni: 4.24 µg/L, Cr: 1.95 µg/L, Ti: 1.68 µg/L, Co: 0.46 µg/L, Cu: 23.31; µg/L, Zn: 220.67 µg/L. After 6 months: Ni: 5.04 µg/L, Cr: 1.01 µg/L, Ti: 9.29 µg/L, Co: 0.32 µg/L, Zn: 168.45 µg/L.	Statistically significant increase in salivary Ti, and statistically significant decrease in Cr and Zn. Non-significant differences difference between both groups.	[257] Jurela et al. (2018)
20 healthy patients + 20 periodontal patients	Brackets (not specified) and NiTi archwires.	Saliva. Sampling 2 months after starting the orthodontic treatment.	Ni. ICP-MS.	Healthy patients: Ni: 182.8 ng/mL, Cr: 6.35 ng/mL. Periodontal patients: Ni: 338.2 ng/mL, Cr: 7.45 ng/mL.	Statistically significant differences in salivary Ni concentrations between healthy and periodontal patients. Inconclusive for Cr.	[258] Amini et al. (2019)
60 patients divided into 2 groups according to the oral health products used (fluorinated vs. nonfluorinated)	SS brackets and NiTi archwires.	Gingival crevicular fluid. Sampling before and after 1 week, 1 month and 6 months after placement.	Ni, Cr, Ti, and Mn.	Nonfluorinated: Before—Ni: 0.49 µg/L; Ti: 0.49 µg/L. 7 days—Ni: 0.52 µg/L; Ti: 0.51 µg/L. 30 days—Ni: 13.42 µg/L; Ti: 40.09 µg/L; Cr: 0.50 µg/L; Mn: 0.50 µg/L. 6 months—Ni: 0.51 µg/L; Ti: 4.80 µg/L; Cr: 0.49 µg/L; Mn: 0.49 µg/L. Fluorinated: Before—Ni: 0.51 µg/L; Ti: 0.51 µg/L. 7 days—Ni: 0.52 µg/L; Ti: 0.50 µg/L. 30 days: Ni: 101.78 µg/L; Ti: 64.69 µg/L; Cr: 12.00 µg/L. 6 months—Ni: 0.51 µg/L; Ti: 0.51 µg/L; Cr: 0.53 µg/L; Mn: 0.48 µg/L.	Statistically significant increase in Ni, Cr, and Ti concentrations at 30 days only. Higher metal content in patients using fluoride-containing oral hygiene products, with statistically significant difference for Ni when compared with patients using nonfluorinated products.	[259] Chitra et al. (2019)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
50 patients + 30 controls	SS brackets and bands; SS and NiTi archwires.	Saliva and serum. Sampling before and during the orthodontic treatment (1 week, 3 months, 1 year, and 1.5 years).	Ni, Cr and Zn. AAS.	Saliva—before treatment: Controls—Ni: 4.33 ppb, Cr: 1.13 ppb, Zn: 10.73 ppb; Patients—Ni: 4.24 ppb, Cr: 1.18 ppb, Zn: 11.8 ppb. Saliva—1.5 years after: Controls—Ni: 5.02 ppb, Cr: 1.27 ppb, Zn: 10.24 ppb; Patients—Ni: 67 ppb, Cr: 30.8 ppb, Zn: 164.7 ppb. Serum—before treatment: Controls—Ni: 8.31 ppb, Cr: 6.18 ppb, Zn: 29.1 ppb; Patients—Ni: 8.46 ppb, Cr: 6.46 ppb, Zn: 28.3 ppb; Serum—1.5 years after: Controls—Ni: 8.47 ppb, Cr: 6.02 ppb, Zn: 30.1 ppb; Patients—Ni: 81.65 ppb, Cr: 35.6 ppb, Zn: 597.16 ppb.	Statistically significant increase in salivary and serum concentrations of Ni, Cr, and Zn between controls and patients. Below toxic levels.	[260] Quadras et al. (2019)
100 patients + 40 controls	Fixed orthodontic appliances (not specified).	Serum. Sampling between 3 weeks and over 18 months.	Ni, Cr, Fe, Cu, Mn, and Zn. ICP-MS.	Controls: Ni: 26.95 µg/L, Cr: 44.45 µg/L, Fe: 200.72 µg/L, Cu: 31.43 µg/L, Mn: 13.75 µg/L, Zn: 32.90 µg/L. Patients: Ni: 61.40 µg/L, Cr: 44.28 µg/L, Fe: 454.92 µg/L, Cu: 55.42 µg/L, Mn: 18.85 µg/L, Zn: 143.70 µg/L.	All ions' concentrations increased in the serum, except for Cr. Ni concentration in serum was dependent on treatment time.	[261] Moghadam et al. (2019)
35 patients	Fixed orthodontic appliances (not specified) involving NiTi and SS archwires.	Saliva and urine. Sampling before and during the orthodontic treatment (3 and 6 months).	Ni and Ti. ICP-OES.		Statistically significant differences in the Ni concentrations in saliva between 3 and 6 months, as well as Ti in urine in the same periods.	[198] Velasco-Ibañez et al. (2020)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
20 patients + 20 controls	SS brackets, bands and archwires.	Saliva. Sampling before and 6–12 months after placement.	Ni. AAS.	Controls: Before: 9.82 ng/mL, After: 10.21 ng/mL. Patients: Before: 9.90 ng/mL, After: 15.83 ng/mL.	Higher but statistically insignificant increase in Ni concentrations in patients when compared with the control group.	[262] Butt et al. (2020)
43 patients divided in groups according to soldering of the lingual arch + 21 controls	Lingual arches composed of SS bands and wires and soldered or welded SS wires.	Saliva. Sampling before and after placement (7, 15 and 30 days).	Ni, Cr, Fe, Cu, Zn, Ag, Cd, and Sn. ICP-MS.	Controls: Ni: 8.0–6.0 µg/L, Cr: 3.5–3.9 µg/L, Fe: 227.9–289.9 µg/L, Cu: 23.1–34.2 µg/L, Zn: 461.0–499.8 µg/L, Ag: 10.1–18.3 µg/L, Cd: 0.6–1.0 µg/L, Sn: 16.6–25.3 µg/L. With lingual arches: Ni: 5.3–34.5 µg/L, Cr: 3.3–4.2 µg/L, Fe: 201.0–314.8 µg/L, Cu: 28.0–40.7 µg/L, Zn: 384.3–963.4 µg/L, Ag: 3.7–20.8 µg/L, Cd: 0.5–1.5 µg/L, Sn: 11.4–27.7 µg/L.	No statistically significant differences for most metallic ions. Below toxic levels.	[263] Schacher et al. (2020)
40 patients divided into 2 groups according to the toothpaste used (nonfluorinated or fluorinated)	Brackets (non-specified) and NiTi archwires.	Gingival crevicular fluid. Before and after placement (7 and 30 days, and 6 months).	Ni and Cr. ICP-MS.	Nonfluorinated toothpaste [ng/mL]: Before: Ni: 0.49, Cr: 0.48; 7 days: Ni: 0.52, Cr: 0.52; 30 days: Ni: 13.4, Cr: 40.6; 60 days: Ni: 0.54, Cr: 4.9; Fluorinated toothpaste [ng/mL]: Before: Ni: 0.52, Cr: 0.52; 7 days: Ni: 0.54, Cr: 0.53; 30 days: Ni: 100.2, Cr: 62.4; 60 days: Ni: 0.52, Cr: 0.52.	Statistically significant increase in Ni and Cr release for patients with prescribed fluorinated toothpastes.	[264] Pritam et al. (2021)
12 patients with fixed appliances + 15 patients with removal appliances	Fixed appliances (NiCr brackets and NiTi archwires); removal aligners (polyurethane).	Saliva. Sampling before and 3 months after starting the treatment.	From Al (Z = 13) to Y (Z = 39). Total reflection XRF.		No significant alterations regarding metals from metal corrosion and inflammatory reactions in patients under dental plaque control.	[265] Zeffa et al. (2021)

Table 6. *Cont.*

Sample Size	Appliances	Matrix and Sampling	Elements and Detection Mode	Mean/Median Concentrations	Main Results	Reference
20 patients	SS brackets (other components were not specified).	Saliva. Before and during treatment (2 weeks, and 1, 4, and 6 months after start).	Ni, Cr, Fe, Ti, and Cu. ICP-OES.	Before treatment: Ni: 3.94 μg/L, Cr:2.37 μg/L, Fe: 45.13 μg/L, Ti: 48.25 μg/L, Cu: 1.53 μg/L. During treatment: Ni: 9.73–34.22 μg/L, Cr: 8.20–17.70 μg/L, Fe: 56.71–99.96 μg/L, Ti: 42.29–62.53 μg/L, Cu: 8.12–25.31 μg/L.	Maximum ion concentrations obtained 1 month after starting the orthodontic treatment. Increase in saliva pH and flow rate. Kinetic model proposed.	[266] Hamadamin (2022)
17 patients	SS brackets, bands, and tubes; NiTi archwires.	Saliva. Sampling before and during treatment (2 days, and 1, 4, and 12 weeks).	Ni, Cr, and Fe. ICP-OES.	Ni: 132–175 μg/L, Cr: 171–192 μg/L, Fe: 826–1023 μg/L.	No statistically significant variations registered throughout the study time.	[116] Fróis et al. (2022)
60 patients	SS brackets; metallic archwires.	Saliva. Sampling 1,3 and 5 years after starting the treatment.	Ni. ICP-MS.	1 week after starting: Ni: 1.25–1.74 ppb; 2 weeks after starting: Ni: 5.76–6.07 ppb; 3 weeks after starting: Ni: 4.32–4.78 ppb.	Significant increase between the 1st and the 2nd sampling time, reportedly linked to the use of hand-held mobile phones.	[267] Rajendran et al. (2023)

Friction always opposes movement between two sliding surfaces under load and may be divided into two modes: Static—when the applied force is still insufficient to induce relative motion—and dynamic—when surfaces are in relative motion (Figure 12). Despite the focus on kinetic friction, the motion of an archwire is hardly ever continuous; therefore, the most likely relevant friction type in orthodontic tooth movement is static friction, opposing any applied force [37,215]. Factors affecting both friction modes include: Geometry and type of archwires, brackets, and ligatures; surface chemical composition and roughness; cleanliness; and lubrication conditions [12,37,68,216,219,268–270]. Moreover, the manipulation of the components when placing and adjusting the orthodontic appliances during the treatment may lead to plastic deformation, wear tracks, and debris. Figure 13 points out these features in a retrieved orthodontic tube after two years of intraoral use.

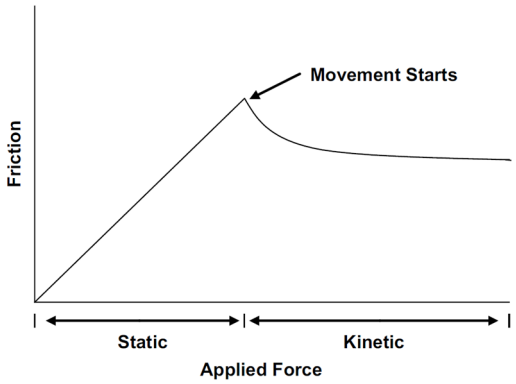

Figure 12. The two types of friction present when a force is applied to an object. Static friction occurs until a certain limit above which the movement starts. Kinetic friction opposes the on-going movement (reprinted from [215], Copyright (2009), with permission from Elsevier).

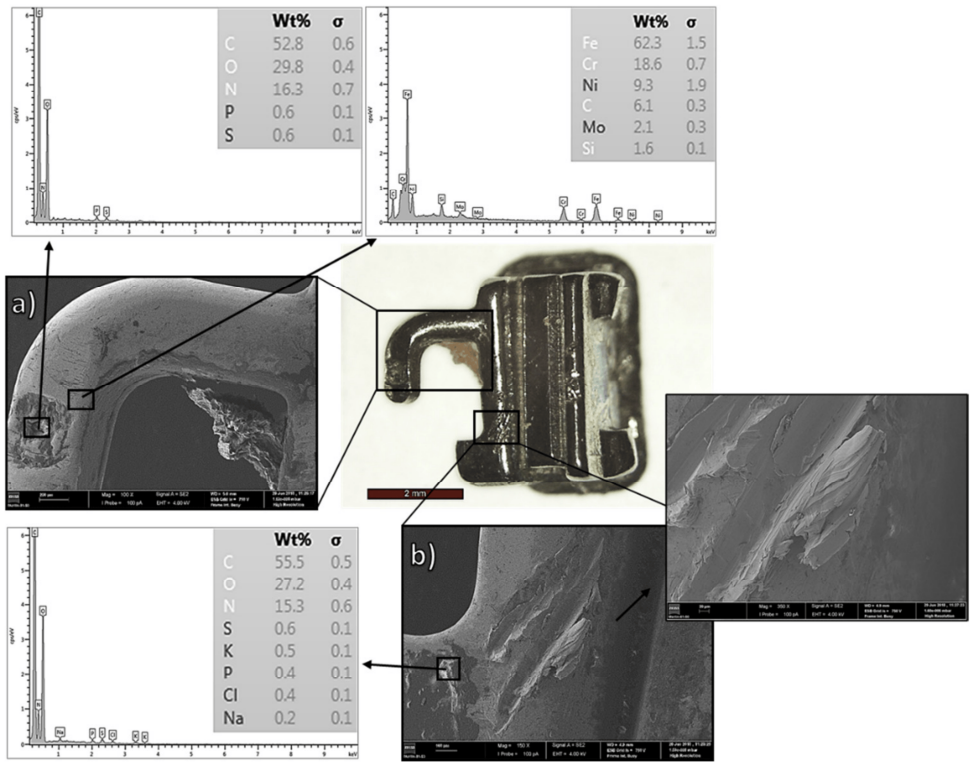

Figure 13. Optical and SEM micrographs of a retrieved SS tube after 2 years of intraoral exposure: (**a**) cavity filled with organic material; (**b**) wear track and debris coupled with the EDS chemical composition (reproduced from [116]).

Saliva is the natural intraoral lubricant by forming a protective pellicle [68–71]—a double layer of proteins [73]—on any material surface and, therefore, reducing the dynamic coefficient of friction [74,271]. While biofilms might have a protective role [66,90,272], microbiological activity most likely contributes to surface degradation by inducing corrosive microcells, rupturing the biofilm, and roughening the appliance surfaces [73]. These effects increase friction, wear, and metallic ions released from the bracket/wire contact pair.

3.3. Oral Hygiene with Fluoride-Based Products

Functional and aesthetic success is essential in orthodontics, but patients must comply with proper oral hygiene during treatment to avoid tooth demineralization and white spot lesions [91,108,273]. Fixed appliances make this task difficult, as the number of oral bacteria related to gingivitis increases shortly after their oral placement [273]. In fact, dental plaque accumulates in several regions (see Figures 7, 9 and 13), namely in the gingival areas or behind the archwires (e.g., on the bracket slots) [28,106,116,137,274]. To fight dental plaque, orthodontists prescribe fluoride-containing toothpastes, mouth rinses, gels, and varnishes to further control its accumulation and growth, enhance enamel integrity, and prevent dental and gingival diseases [72,91,275].

The downside of using these fluorides is the increased corrosion susceptibility of metallic alloys [29,42,264,276–282]. Fluoride ions (F^-)—combined with mechanical brushing—easily degrade the protective oxide layers of both SS and Ti-based alloys (see Equations (8) and (9)), increasing localized and general corrosion, promoting metallic ions release [276,283], and negatively impacting their mechanical and surface properties [41,42,277,279,282,284–286]—especially at low pH [104,118] and under the simultaneous presence of chloride ions [287].

Walker et al. [41] reported reduced unloading mechanical properties of SS and β-Ti archwires when exposed to neutral or acidulated prophylactic fluoride gels, which may prolong the orthodontic treatment time. On the other hand, Sufarnap and colleagues [56] reported an increase in both surface roughness and Ni and Cu release from Cu–NiTi archwires in NaF solution in vitro without a significant change in the deflection force.

Corrosion of SS bands and brackets [117,118,288,289] also increases in the presence of fluoride ions. Chantarawaratit and Yanisarapan [289] argued that acidulated phosphate fluoride gel should not be used in patients wearing fixed metal-based orthodontic appliances.

Since these SS components are the support for NiTi or Cu-NiTi archwires, galvanic coupling risk increased during the 1st orthodontic treatment phase (leveling/aligning) with possible mechanical and/or biocompatibility-related adverse consequences [108,112,284]. Figure 14 evidences the risk of galvanic coupling between dissimilar bracket/archwire pairs (concerning their alloys' composition), showing different in vitro metallic ion release profiles, according to the three solutions used. Synthetic F^--free Fusayama–Meyer saliva—widely used in in vitro studies—showed lower aggressiveness than the commercial mouthwashes containing fluoride [108].

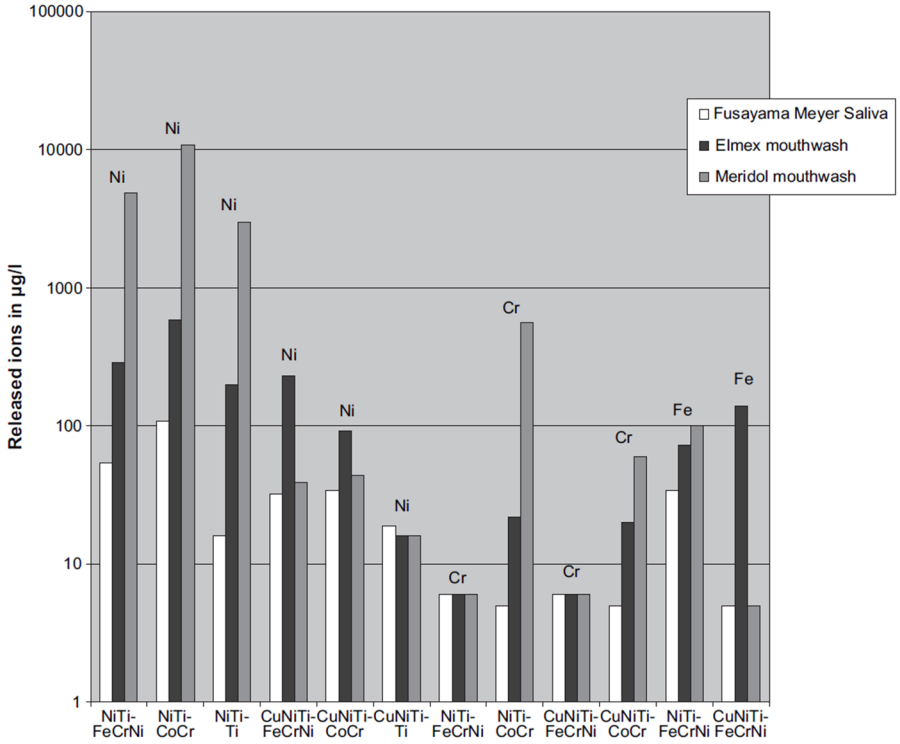

Figure 14. Metal ions (in µg/L) released by wire-bracket combinations in three different solutions (reprinted by permission of Oxford University Press on behalf of the European Orthodontic Society, from [108]; permission conveyed through Copyright Clearance Center, Inc.).

4. Orthodontic Alloys Modification

The main topic of this chapter is to briefly point out the current mitigation strategies to reduce the metal corrosion susceptibility of orthodontic appliances, including existing and emerging approaches for surface and bulk alloy modifications.

4.1. Non-Metallic Components

One way to reduce the release of metallic ions due to intraoral corrosion is to replace metallic-based alloys with non-metallic materials. Several examples of Ni-free substitutes for common orthodontic devices and components are listed in Table 7. However, existing or under development non-metallic alternatives for orthodontic appliances are more focused on satisfying patients' increasing aesthetic demands rather than on clinical concerns. Polymeric brackets—made of polycarbonate (PC), polyurethane (PU), or polyoxymethylene (POM); ceramic brackets—as those made of mono or polycrystalline aluminum oxide (Al_2O_3); non-metallic brackets with and without metallic slots [10,11,253,290–293]; and non-metallic archwires—produced from thermoplastic polymer [294], polyphenylene (PP) [295], poly-ether-ether-ketone (PEEK) resin [296], and fiber-reinforced polymers [34,297,298]—are some examples. A systematic literature review on transparent orthodontic archwires was prepared by Mikulewicz and colleagues [299], while J. Russel [290] reviewed aesthetic orthodontic brackets. Table 8 summarizes some of the current commercially available non-metallic aesthetic brackets and archwires.

Table 7. Ni-containing orthodontic materials and the corresponding Ni-free substitutes (used with permission of The Edward H. Angle Education and Research Foundation, from [28]; permission conveyed through Copyright Clearence Center, Inc.).

Category	Material	Ni-Free Substitute and Modifications
Standard appliances	Brackets.	Ni-free SS, ceramic, plastic, Ti, gold-plated or coated with other precious metals (Pd, Pt) brackets.
Treatment utilities	Bands. SS archwires.	Gold-plated bands. No alternative currently available; development of polymeric wires in progress.
Mechanic helpers	CoCrNi archwires. Sliding yokes, transpalatal. and lingual arches.	No alternative currently available β-Ti (TMA), plastic or inert metal (gold) coatings of wire segments.
Miscellaneous helpers	SS ligatures. Kobayashi hooks. Coil springs.	Teflon-coated ligatures. Teflon-coated Kobayashi hooks; Ni-free brackets with hooks. Elastomeric ligatures.
Fixed expansion appliances	SS appliances (Quad Helix). Rapid palatal expander. SS headgear. NiTi spring screws.	β-Ti (TMA) wires for Quad-Helix. Teflon-coated SS facebow. No alternative currently available.
Removable appliances	SS components of Hawley appliances and variations.	Plastic or elastic retainers; elastic positioners or acrylic splints invisagen^{TM} technique.
Complex therapeutic interventions	Orthognathic surgery lag screws and plates. Distraction osteogenesis apparatus.	Resorbable polylactic-polyglycolic lag screws and plates. No alternative currently available.

Multiple concerns [11,300] still limit clinicians' acceptance and widespread use of alternative aesthetic solutions [290,301]. For instance, polymeric-based brackets may experience hardness reduction [291], undergo discoloration and staining from food dyes and sterilization procedures [302,303], release highly cytotoxic substances (e.g., bisphenol A [BPA]) [304], or have difficulty sliding against metallic archwires [11]. Similarly, ceramic brackets may also stain and discolor [305] during orthodontic treatment, in addition to their brittleness and high susceptibility to fracture [290,306,307], increased friction [308] and more severe archwire notching [218], higher pain during treatment [309], and enamel damage [11]. In a recent in vitro study concerning bracket genotoxicity [194], the authors even argue that ceramic brackets are more genotoxic than conventional SS or CoCr alloy ones. Non-metallic, aesthetical archwires present limitations as well. The foremost drawbacks—crazing formation, plastic deformation, and lower deliverable forces when bended [297,310] in addition to noticeable color change [311]—have limited their clinical widespread use.

Table 8. Non-metallic available aesthetic brackets and archwires [10,11,253,290,293,297–299,310].

	Brackets	Archwires
Polymeric	Polyurethane (PU). Polycarbonate (PC). Polyoxymethylene (POM). Fiberglass-reinforced PC.	Polyethylene terephthalate (PET) polytetrafluoroethylene (PTFE). Polyetheretherketone (PEEK). Fiber-reinforced polymer composites, and 3-layered wire (SiO_2/silicone resin/nylon).
Ceramic	Alumina. Zirconia.	

4.2. Nominal Composition Optimization

Another approach to decreasing intraoral corrosion susceptibility is to adjust the bulk nominal composition of the alloys by adding or subtracting certain elements [95,312]. As already mentioned in Section 2, Ni stabilizes the austenitic phase of SS and increases the

alloy's resistance to corrosion and oxidation [10], namely against non-oxidizing acids [127]. However, the abovementioned clinical concerns make these alloys undesirable. Ni-"free" SS alloys (maximum of 2% Ni) were pointed out as viable alternatives to allergic orthodontic patients to this transition element [313]. The main development involves, at least, the presence of another element, such as Mn [10], in the SS nominal composition. Nevertheless, the final microstructural, mechanical, and corrosion resistance properties of the resulting steel can be compromised [36]. Additionally, Ni-"free" brackets/tubes are more cytotoxic than Ti-based ones [312] and may still provoke oral allergic reactions [314], even though conventional SS alloys are less biocompatible [304]. Another alternative approach is appropriately described by Eliades et al. [28] concerning the replacement of the most commonly used bracket SS alloy—316L AISI—by the duplex SS 2205 grade, which contains less Ni nominal content (Table 3).

To mitigate the nickel impact on oral health, the high Ni-containing alloys (TiNi) have been chemically optimized by adding Ag or Cu. The ternary TiNiAg alloys show similar mechanical properties and cytotoxicity effects as the binary TiNi ones and are more effective in reducing bacterial adhesion [315], but up to a certain Ag content limit [316] (reportedly 0.5–1.5 wt.% by Oh et al. [317]). Newer Cu–NiTi archwires [34] provide thermal activation and increase their resistance to permanent deformation. However, such alloys cannot be easily welded, are expensive, have low formability and high friction against SS brackets, and may fracture when bent over sharp edges [10,42,53]. New composite archwires (CAW), produced by NiTi and SS segments laser-welded with Cu, also seem to be promising for orthodontic treatments due to their excellent mechanical properties. Nevertheless, the resulting Cu interlayer could corrode in in vivo conditions, leading to biocompatibility and performance complications [318].

4.3. Manufacturing Processes

Different manufacturing processes lead to different final microstructural and physicomechanical properties that can influence the overall corrosion resistance of metallic appliances. Brackets, tubes, and bands, for instance, can be made by casting and/or machining (milling) separate metallic pieces (e.g., the base and the wings, as shown in Figure 15). These parts are joined by soldering or welding [319]. Ag-based brazing alloys were popular and are still widely used [320], but these solders may dissolve or induce galvanic coupling inside the oral cavity, releasing cytotoxic ions [111,319,321,322]. Alternatives include the use of Au-brazing alloys, which induce galvanic corrosion of the less-noble SS following the Ag case, or Ni-based brazing alloys, with no available data [98,319] but undesirable due to their toxicity. Laser welding, a more recent and preferable advanced manufacturing technique, allows joining without brazing alloys [95,192,322], with lower corrosion susceptibility [323], even though large gaps in the joints may be a disadvantage for Ti-based brackets [319].

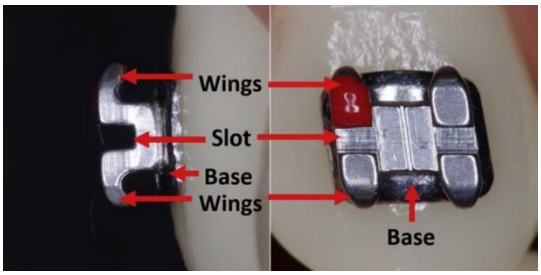

Figure 15. Identification of the bracket parts: wings, slot, and base (reprinted from [319], Copyright (2017), with permission from Elsevier).

Metal injection molding (MIM) is nowadays a popular manufacturing technique [11,319] to provide single-piece metallic orthodontic components with superior surface finishing in a cost-effective manner. Advantages over conventional casting/welding brackets include the absence of galvanic coupling between the base and the wings, the reduction of oral metallic

ion release, and lower wear when used with NiTi archwires contacts [98,242,319]. However, internal porosity and possibly different biocompatibility and electrochemical behaviors of well-known alloys are some concerns that motivate further optimization [98,242,319].

Important developments in orthodontic appliance manufacturing include the production of functionally graded archwires (that apply different triggering forces depending on the location, accomplished by heat treatments) [324] and customized pieces by additive manufacturing [325–327], which may lead to less prolonged and more effective orthodontic treatments.

4.4. Surface Modification and Coatings

Another important optimization route for orthodontic alloys is surface modification and/or coatings deposition, preserving their best features, that is, their bulk properties. Corrosion most likely develops on bearing surfaces (Section 2) rather than in the bulk. Hence, different surface finishes can greatly impact the alloys' corrosion performance [95,328]. Mechanical polishing, for example, can reduce surface defects, leaving a smooth topography [329]. According to Hunt et al. [96]—who polished metallic archwires without impacting surface hardness—this process seemed detrimental or indifferent to CoCr, SS, and β-Ti archwires exposed to a corrosive medium (0.9% NaCl solution), but polished NiTi archwires presented higher corrosion resistance than its pristine condition [96]. Electropolishing is a common treatment to enhance both the appearance and corrosion resistance of the components, but galvanic corrosion cells may be produced between polished and unpolished regions [95]. Ion implantation is also a commercially used surface treatment of NiTi archwires [329,330] to reduce friction when compared with uncoated ones. However, Wichelhaus et al. [329] showed that this effect only occurred at the beginning of the orthodontic treatment, alerting them to the risks of recycling these components.

Current progress is also devoted to thin film/coating deposition with new and unique properties—a versatile approach to improving the final surface properties [39]. Within this regard, the flowchart presented in Figure 16 condenses the biomaterial surface properties that can be adjusted by plasma synthesis, as proposed by Chu et al. [331]. Without error, it can be generalized to a variety of methods and technologies available for biomedical applications, including orthodontics.

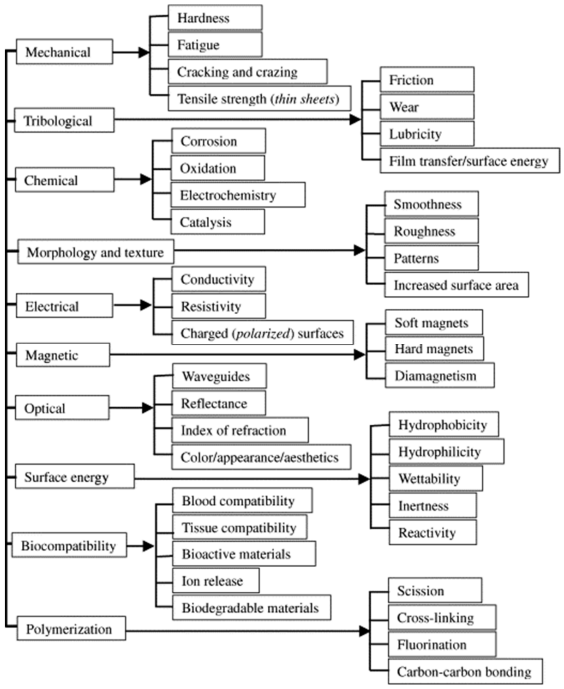

Figure 16. Summary of biomaterials surface properties (reprinted from [331], Copyright (2002), with permission from Elsevier).

Due to the high importance of coatings' deposition in tailoring the surface of orthodontic bioalloys, the authors opted to present and develop this topic separately in the following chapter (Section 5).

5. Protective Coatings in Orthodontics

This chapter aims to present the multitude of coatings available in the literature that have been deposited onto real fixed orthodontic appliances (the widely used archwires, brackets, and bands) to improve features such as corrosion, friction, fretting, biocompatibility, and antibacterial activity. For that, the results of the current review will be presented considering the three main groups: metal-, polymeric- and ceramic-based coatings (Tables 9–11). Several industrial deposition techniques can be used, as reviewed by Arango et al. [39] and presented in Figure 17, some of them which are applied in orthodontics.

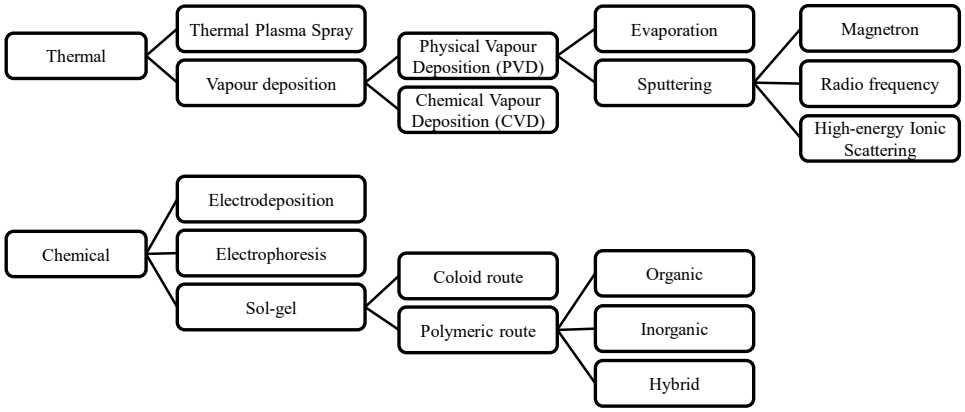

Figure 17. Classification of coating processes used at industrial level (adapted from [39]).

5.1. Metal-Based Coatings

Due to their higher corrosion resistance in addition to their aesthetic and pleasant color, metallic-based coatings have been deposited onto fixed orthodontic appliances, as presented in Table 9. Most pure metal coatings are devoted to rhodium (Rh), gold (Au), and silver (Ag). Titanium (Ti) and zinc (Zn) films were also included in this summary due to their excellent biocompatibility.

5.1.1. Transition Metal-Based Coatings

- Rhodium (Rh):

Rhodium-coated archwires—usually as bimetallic Rh–Au coatings [332–334]—are available on the market due to their pleasant color and superior corrosion resistance, without compromising the mechanical properties of the bulk alloys [253,310,335–338].

Katić and coauthors [332,339–341] compared the corrosion resistance of the uncoated NiTi archwires with those nitrified and coated with Rh. Surprisingly, electrochemical tests showed that corrosion susceptibility increased in artificial saliva, being highest for Rh-coated archwires: The heterogeneous layers induced galvanic coupling between the noble metals (Au and Rh) and the NiTi alloy, causing pitting corrosion and pronounced Ni release, as well as aesthetical appearance degradation [339,340]. Other researchers compared different types of coated and uncoated archwires, finding the worst corrosion resistance for the Rh-coated ones [61,342]. However, when fluoride-based prophylactic agents were weakly added during a 4-week immersion test in artificial saliva, ionic release from Rh-coated archwires decreased with increasing F ionsconcentration [332].

Nsaif et al. [343] found a protective effect of Rh-based coatings against an acidulated phosphate fluoride agent without compromising the overall mechanical properties of the archwires. Mlinaric et al. [279] demonstrated that the alteration of the properties of Rh-coated NiTi archwires depends on the type of oral antiseptics used. In turn, Osmani

and colleagues [338] advised that such archwires should be avoided if oral hygiene is compromised due to the higher release of metallic ions.

The use of probiotic supplements—recommended to prevent caries and gingivitis during the fixed orthodontic treatment—also negatively impacts the corrosion resistance of Rh-coated NiTi archwires in artificial saliva: Both general and localized (pitting) corrosion increased, with precipitation of corrosion products on the surfaces [344,345]. However, a simple 1-month immersion test of Rh-coated SS and NiTi archwires in probiotic-containing artificial saliva suggested a decrease in corrosion susceptibility, possibly due to a benign anticorrosion effect of the early-formed biofilms [346]. Since mature biofilms will more likely promote MIC, the in situ higher accumulation of microorganisms for Rh-coated NiTi archwires reported by Lima et al. [347] may be a demerit result. Other researchers disagree [63], reporting that: (i) commercial Rh-coated NiTi archwires can significantly decrease *S. mutans* bacteria adhesion in vitro by altering the apparent surface free energy; and (ii) Au coating on SS archwires did not significantly influence the adhesion of *S. mutans* due to similar apparent surface free energy as uncoated SS [63].

Usui et al. [348] compared the kinetic and static frictional properties of both uncoated and Rh-coated SS archwires, under dry and wet conditions, against a ceramic bracket. In all tests, coated wires generated significantly higher kinetic and static frictional forces than the uncoated ones—more than 46% higher. Still, three-point bending test results indicated equivalent flexural properties. The bending effect can, however, increase the surface roughness of NiTi archwires coated with Rh-based materials [333]. Albawardi and colleagues [349] concluded that Rh-coated β-Ti archwires are not ideal to decrease friction against brackets for angulations up to 10° when compared to uncoated β-Ti or SS wires.

The color stability of commercial Rh-coated NiTi archwires has also been investigated by immersion in a coffee staining solution. While Alsanea and Shehri [335] found just a slight discoloration after 4 weeks, Ramasamy and colleagues [350] reported an extremely marked color change after 3 weeks of wetting.

- Gold (Au) and Platinum (Pt):

Gold and platinum are two noble metals that are widely used in many medical applications due to their bioinertness and notable corrosion resistance [351,352]. Despite their high cost, Au coatings are commercially available in orthodontics [62,64]. Surprisingly, Toy et al. [62] found that Au-plated SS brackets "remarkably decreased cell proliferation of HGFs" (human gingival fibroblasts), possibly related to an increase in in vitro corrosion during the test caused by surface defects of the substrate alloy. Conversely, other tested brackets (uncoated, made of metallic, ceramic, and composite materials) increased cell proliferation [62]. In other research work, Ito et al. [61] evaluated the coating loss of Au-coated SS wires after acid immersion (35% hydrochloric acid, pH = 1.1), wire ending, and mechanical brushing. While the Au coating resisted mechanical brushing and effectively prevented the release of metallic ions during immersion, wire bending showed signs of cracks and delamination of the coated wire [61]. Krishnan et al. [64] studied several coated and uncoated NiTi archwires and found that the breakdown potential of Au-coated ones is only surpassed by PTFE-coated wires. Regarding Pt-based coatings, Khonsari et al. [353] deposited Pt nanoparticles (NPs) on NiTi archwires by an electrochemical method, achieving adherent and homogeneous layers with a suitable tooth-like color.

- Silver (Ag):

Silver is recognized as one of the oldest antibacterial agents known, dating back to ancient times, at least by ~1500 B.C. [354,355]. In orthodontics, Ag-based coatings have been used to take advantage of this feature, that is, to reduce both bacterial adhesion and biofilm growth. Hence, periodontal diseases, tooth decay, and demineralization [356] have been prevented during orthodontic treatments.

Mhaske et al. [357] deposited pure Ag onto two types of archwires (NiTi and SS) by thermal vacuum evaporation. In addition to its antimicrobial effect, the 10 nm thick film effectively reduced the in vitro adhesion of *L. acidophilus* on both wire types, and,

therefore, the total weight increase due to biofilm accumulation was lower: 35.5 and 20.5% for uncoated SS and NiTi wires, respectively, and less than 4.5% for Ag-coated wires [357]. Ghasemi et al. [358] reported that 60 and 100 μm thick Ag films deposited by physical vapor deposition (PVD) onto SS brackets were able to reduce the *S. mutans* counts right after 3 h of incubation.

Recently, an in situ study analyzed the initial biofilm formation (up to 48 h) onto an austenitic Ni-free bracket coated with Ag by using 3 distinct production processes: PVD, galvanic, and plasma immersion ion implantation and deposition (PIIID) processes [359]. The results showed statistically significant lower biofilm volume and surface coverage for all thin-film types in comparison to uncoated samples, but not between modified samples. All coatings showed antibacterial properties, with the PIIID-modified bracket displaying the highest increase in the dead/alive cell proportion.

An in vivo study lasting up to 75 days was conducted by Metin-Gürsoy et al. [360] to evaluate the antibacterial properties and the ion release from nano-Ag-coated SS brackets. The PVD-coated brackets placed on the mandibular incisors of Wistar Albino rats effectively inhibited *S. mutans* growth, which reduced caries development on the smooth surfaces. Concerns regarding the biocompatibility of Ag-based coatings for orthodontic applications previously motivated the same authors [356] to perform a 60-day animal histological experiment. Encouraging findings indicated that uncoated and PVD-nano-Ag-coated standard brackets implanted in healthy rats have similar biocompatibility behaviors. Nevertheless, the authors found brown-black silver granules in the adjacent tissues. Further studies are required to assess an eventual negative biological impact.

The research work conducted by Espinosa-Cristóbal et al. [361] described the influence of silver nanoparticles' (Ag-NPs) size on the activity of *S. mutans*. The coatings were chemically deposited on both SS brackets and archwires. The obtained results showed that coatings possessed good antimicrobial and anti-adhesion properties against this bacterial strain, especially for smaller-sized Ag-NPs (diameter of ~8.1 nm). Similarly, other authors [362] reported a significant decrease in the activity of *S. mutans* and *L. acidophilus* after a 24–48 h incubation with Ag-NPs-coated segments of SS orthodontic bands. Ag-NP coatings provide antimicrobial properties to SS brackets [48], bands [363,364] and archwires [365], namely against *S. mutans*, *S. sobrinus*, *C. albicans*, and *L. acidophilus* [48,364,365]. Coated NiTi archwires induced a more than 90% decrease in the adhesion of *S. sanguinis* and *L. salivaris* [366]. Zeidan et al. [367] reported an Ag-NPs coating (deposited on SS brackets by the thermal evaporation method) with an antibacterial effect against *S. mutans* and *L. acidophilus* over a period of 3 months of incubation. According to an interesting work by Anand et al. [368]—who synthesized Ag-NPs from plant extracts—the well-known antibacterial effect of Ag-NP coatings can be further improved by simultaneously adding TiO_2-NPs to functionalize orthodontic archwires. These coatings "*showed an enhanced antibacterial effect against Multi-Drug Resistant (MDR) bacteria*" [368].

Since corrosion and abrasion impact the intraoral performance of coatings, Ryu et al. [369] added Pt (up to 7%) to Ag coatings by PVD. The goal was to increase chemical stability. From the potentiodynamic polarization tests, the resulting AgPt-coated SS substrates showed higher corrosion resistance than the single Ag-coated or uncoated samples. In addition, all coatings diminished *S. mutans* and *A. actinomycetemcomitans* growth by up to 60%. Simultaneously, the cytotoxicity due to silver ion release was statistically insignificant when tested with human gingival fibroblasts. The authors [369] claim that brackets and archwires can be coated with these binary Ag–Pt materials, even though only flat substrates were used in their studies.

Whereas the (micro)biological impact is very important in the oral cavity, the Ag-based coatings must not compromise the surface tribological properties, especially the frictional behavior. Thermal vacuum evaporation-synthesized Ag coatings (~10 nm thick) onto two types of SS archwires did not affect their frictional resistance against SS brackets [370]. Reportedly, friction in the SS bracket/archwire combination decreases when at least the wire

is Ag-coated [371]. Nevertheless, thick Ag-electroplated SS brackets (8–10 μm) significantly increased both surface roughness and friction forces against SS and NiTi archwires [372].

Shirakawa, Usui and colleagues [373,374] deposited tooth-colored Ag coatings on SS wires by eletropolishing. After 1 week of in vivo use with metallic brackets, no significant peeling was observed; however, the part of the wire in contact with the bracket slot turned black after 2 days [373].

- Titanium (Ti):

Titanium is scarcely used as a surface material in fixed orthodontics. To the authors' knowledge, only two research works (see Table 9) were found concerning fixed orthodontic appliances [375,376]. Ozeki and colleagues [375,377] coated Nitinol archwires with a 1 μm thick Ti layer by magnetron sputtering. This surface modification effectively reduced the Ni release in physiologically saline immersion tests up to 8 weeks (~5 times lower than uncoated wires). On the other hand, the superelasticity of the NiTi alloys decreased by almost 10.4% in comparison to uncoated wires, and the three-point bending tests (for 2 mm deflection) revealed a load decrease of 39% (from 4.18 to 2.55 N). Despite the decline in mechanical behavior, Ozeki et al. suggested that the performance of the sputter-coated archwires was not clinically compromised. In fact, when used in vivo for 4 weeks, both coated and uncoated archwires showed no inflammation signs, and little coating exfoliation—attributed to the use of pliers—was documented. Yet, the total sample size was very small: Only 5 patients [375]. Anuradha et al. [376] immersed NiTi archwires coated with sputter-deposited Ti (3–5 μm thick) in artificial saliva for 30 days. The results showed a well-adherent and smooth coating that remained mechanically stable over the test duration, without Ni or Ti detection in the artificial saliva solution.

- Zinc (Zn):

Zinc is an essential trace element [378–380], and has been used in medicine and dentistry due to its antibacterial effects, bioactivity/biodegradability, and good mechanical properties [381–384]. Karandish et al. [381] first reported the study of PVD-deposited Zn coatings on SS archwires. According to the obtained results, these coatings can improve both tensile and load-bending strength. In addition, the deposited Zn layers successfully reduced the friction resistance for the bracket/wire angulation of 10° by a factor of ~50%.

5.1.2. Metal-Based Oxides, Nitrides and Carbides Coatings

The addition of interstitial elements—such as oxygen (O), nitrogen (N), carbon (C), or their combinations—to transition metals to form binary, ternary, or quaternary systems is a practical and common method to enhance the hardness performance of protective coatings, mainly those used in mechanical/tribological applications [385]. In orthodontics, Ti-based coatings have a special position. Although the authors did not account for any research work involving the Ti–C system in fixed orthodontic appliances, the same cannot be said concerning those of Ti–O and Ti–N, as can be seen in Table 9. In addition, some research on binary Zr–O, Al–O, Zn–O, W–C/N, and Cr–C systems will also be considered.

- Ti–O System:

The Ti–O system, particularly the titanium oxide phase TiO_2, is nowadays commercially available for coated brackets and archwires [64]. This surface material is well recognized as inert and more stable than the native oxide layer formed onto Ti-based alloys, preventing the degradation of the alloys and the subsequent oral metal release [386]. Electropolishing, followed by sterilization and thermal passivation, not only smooths the surface of bulk metals but also induces the formation of a desirable TiO_2 top layer on the NiTi alloys [386].

Espinar et al. [387] heat-treated NiTi archwires to promote oxygen diffusion, forming a highly-adherent TiO_2 passive film whose thickness was proportional to both treatment temperature and time. The resulting Ni-depleted surface (up to approximately 20 nm depth) exhibited an inferior friction coefficient against 316 SS disks in comparison to untreated

archwires: CoF = 0.40 and 1.53, respectively. This oxidation treatment can, therefore, make the bracket/archwire sliding easier and prevent Ni-induced allergy and cytotoxicity since the release of metallic ions decreased.

Horiushi et al. [388] heat treated electrolytically grown TiO_2 on NiTi alloys. They intended to develop a rutile crystalline phase and achieve photolytic activity when exposed to ultraviolet radiation (UV-A light, 315–400 nm), which could provide antibacterial activity. Other researchers explored the photocatalytic effect of TiO_2-based coatings deposited on metallic substrates by sol–gel [389–392] and by magnetron sputtering [393–398] methods. In particular, surface functionalization of SS and NiTi archwires [389,390] and SS brackets [395,397] showed effective decays in bacterial adhesion and survival rates of representative oral microorganisms, namely for *P. gingivalis*, *S. mutans*, *L. acidophilus*, and *Candida albicans*, when exposed to UV-A radiation. Reactive oxygen species (including hydroxyl radicals) decompose surface organic molecules of those microorganisms and damage cell walls, allowing the control of dental plaque growth [395]. On the contrary, Awata et al. [391] found no statistically significant difference in the amount of bacterial adhesion after coating 316L SS disks via sol–gel. Zhang et al. [399] reported that nano-TiO_2 coatings deposited on metallic brackets failed to show antibacterial activity up to 240 min in the dark. However, a nano-Ag–TiO_2 system demonstrated antibacterial activities of ~80–94% after 20 min in the dark, depending on the bacteria strain.

Fatani et al. [398] added silver to sputter-deposited photocatalytic TiO_2 coatings. The results confirmed the beneficial combination effect of this system: When compared with uncoated and Ag or TiO_2-coated metallic SS brackets, Ag–TiO_2 coatings showed lower *S. mutans* and *P. Gingivalis* adhesion and growth and biofilm development without cytotoxicity effects on human gingival fibroblast (HGF) cells. Kielan-Grabowska et al. [400] produced TiO_2 layers containing Ag-NPs via the sol–gel dip-coating method on SS 316L orthodontic wires. The results revealed a significant decrease in the overall corrosion resistance of the coated samples, even though they might possess antimicrobial properties [400,401].

While TiO_2 photocatalytic activity requires UV-A light, magnetron-sputtering-deposited and annealed N-doped Ti oxide films on SS brackets showed similar anti-adherent and antibacterial activity when exposed to visible light [393,394]. A 60-minute irradiation period was enough to prevent in vitro *S. mutans* growth for up to 3 months [393]. Nitrogen addition also seems beneficial to diminish the bracket/archwire friction [402].

Ghasemi et al. [358] deposited 60 and 100 μm thick TiO_2 layers on SS brackets by PVD. Results showed that such oxide coatings effectively improved surface roughness and bacterial growth but failed to reduce friction against archwires. Other authors also reported an inhibition of bacterial adhesion on PVD-deposited TiO_2 layers on SS orthodontic components [403].

Jung and colleagues treated both TMA and NiTi archwires with a plasma electrolytic oxidation (PEO, or micro-arc oxidation—MAO) method [404]. The 20–30 μm thick ceramic coating had a pleasant appearance and a higher biocompatibility and could improve the wear resistance of the NiTi archwire [405]. However, wire mechanical bending tests revealed the formation of defects in the coatings, cracking and chipping, and releasing sharp-edge particles that may harm teeth enamel or soft tissues [404].

Campeol et al. [406] reported that delamination of modified plasma-oxidized NiTi wires only occurs at high processing temperatures (>200 °C). The researchers prevented in vitro Ni release and delamination from substrates with an optimal 75–100 nm thick TiO_2 layer at ~135 °C by using a simplified plasma-assisted method. Moreover, the superelasticity of plasma-treated Nitinol wires increased by 8.6% (at 13% strain) when compared with untreated metallic samples [406].

Supriadi et al. [407] suggested that a previous electropolishing step smooths and cleans the surface of 17–4 PH SS orthodontic brackets and consequently increases the adherence of magnetron-sputtered TiO_2 coatings to the metallic substrates. Sol–gel-synthesized TiO_2 (300–400 nm) directly formed on 316L SS plates by Fu et al. [408] showed long and thin cracks and just slightly increased surface hardness. The solution involved the deposition of

a thick TiN interlayer (~8 μm) that prevented the formation of large cracks and significantly increased the hardness (from 280 HV to 810 HV). The main disadvantage reported by the researchers was the decrease in toughness due to surface oxidation, with variable corrosion behavior in a 0.9% saline solution depending on the annealing temperature.

Regarding new composite archwires, Liu et al. [409] used magnetron sputtering to attain TiO_2 and N-doped TiO_2 coatings. They reported an 87% reduction in the antibacterial activity against *S. mutans* of N-doped TiO_2 films (~1.34% of N), in contrast to the undoped films (6% decrease only). The N addition was also beneficial to the in vitro biocompatibility and to the corrosion resistance of bulk CAW, enhancing both performances [409].

For further reading, an interesting meta-analysis was recently published by Solanki et al. [410] concerning in vitro studies using TiO_2 coatings on orthodontic brackets. The notable antibacterial effect with low cytotoxicity on eukaryotic cells was highlighted.

Table 9. Overview of metal-based coatings deposited on different orthodontic appliances and substrates. PVD: physical vapor deposition; PIIID: plasma immersion ion implantation deposition; PEO: plasma electrolytic oxidation; CVD: chemical vapor deposition.

Coatings	Substrate Materials	Deposition Methods	Literature References
Transition Metal-based Coatings			
Rh	NiTi archwires	*Commercial*	[339] Katić et al. (2014)
Rh	NiTi archwires	*Commercial*	[340] Katić et al. (2014)
Rh	NiTi archwires	*Commercial*	[63] Kim et al. (2014)
Rh	NiTi archwires	*Commercial*	[344] Trolic (2017)
Rh	NiTi archwires	*Commercial*	[332] Katić et al. (2017)
Rh	NiTi archwires	*Commercial*	[253] Lages et al. (2017)
Rh	NiTi archwires	*Commercial*	[310] Matias et al. (2018)
Rh	NiTi archwires	*Commercial*	[411] Asiry et al. (2018)
Rh	SS archwires	*Commercial*	[348] Usui et al. (2018)
Rh	NiTi archwires	*Commercial*	[341] Katić et al. (2018)
Rh	NiTi and SS archwires	*Commercial*	[343] Nsaif et al. (2019)
Rh	NiTi archwires	*Commercial*	[335] Alsanea and Shehri (2019)
Rh	NiTi archwires	*Commercial*	[346] Trolic et al. (2019)
Rh	NiTi archwires	*Commercial*	[347] Costa Lima (2019)
Rh	NiTi archwires	*Commercial*	[345] Trolic et al. (2019)
Rh	NiTi archwires	*Commercial*	[279] Mlinaric et al. (2019)
Rh	NiTi archwires	*Commercial*	[337] Batista et al. (2020)
Rh	NiTi archwires	*Commercial*	[336] Pinzan-Vercelino et al. (2020)
Rh	NiTi archwires	*Commercial*	[350] Ramasamy et al. (2020)
Rh	NiTi archwires	*Commercial*	[412] Madasamy et al. (2021)
Rh	NiTi archwires	*Commercial*	[338] Osmani et al. (2022)
Rh	NiTi archwires	*Commercial*	[342] Amorim et al. (2022)
Rh	SS archwires	*Commercial*	[49] Ito et al. (2022)
Rh	β-Ti archwires	*Commercial*	[349] Albawardi et al. (2022)
Rh-Au	NiTi archwires	*Commercial*	[334] Iijima et al. (2012)
Rh-Au	NiTi archwires	*Commercial*	[333] Albuquerque et al. (2017)
Au	SS archwires	*Commercial*	[63] Kim et al. (2014)
Au	NiTi archwires	*Commercial*	[64] Krishnan et al. (2014)
Au	SS brackets	*Commercial*	[62] Toy et al. (2014)
Au	SS archwires	*Commercial*	[49] Ito et al. (2022)
Pt-NPs	NiTi archwires	Electrochemical deposition	[353] Khonsari et al. (2011)
Ag	SS and NiTi archwires	Thermal evaporation	[357] Mhaske et al. (2015)
Ag	SS brackets	Electroplating	[372] Arash et al. (2015)
Ag	SS brackets	Magnetron sputtering	[398] Fatani et al. (2017)
Ag	SS brackets	PVD	[358] Ghasemi et al. (2017)
Ag	SS wires	Electroplating	[374] Usui et al. (2017)
Ag	SS wires	Electroplating	[373] Shirakawa et al. (2017)
Ag	SS archwires	Thermal vacuum evaporation	[370] Shah et al. (2018)
Ag	SS brackets	Galvanic, PVD, and PIIID	[359] Meyer-Kobbe et al. (2019)
Ag	SS brackets and archwires	Thermal vacuum evaporation	[371] Shah et al. (2023)
Nano-Ag	SS brackets	PVD	[356] Metin-Gürsoy et al. (2016)
Nano-Ag	SS brackets	PVD	[360] Metin-Gürsoy et al. (2017)
Nano-Ag	SS brackets and archwires	Chemical deposition	[361] Espinosa-Cristóbal et al. (2018)
Ag-NPs	SS bands	Thermal evaporation	[363] Prabha et al. (2016)
Ag-NPs	SS bands	Thermal evaporation	[362] Bindu et al. (2019)
Ag-NPs	SS archwires	Hydrothermal synthesis	[365] Gonçalves et al. (2020)

Table 9. *Cont.*

Coatings	Substrate Materials	Deposition Methods	Literature References
Ag-NPs	SS and Co–Cr brackets	*Commercial*	[48] Jasso-Ruiz et al. (2020)
Ag-NPs	NiTi archwires	Electrodeposition	[366] Gil et al. (2020)
Ag-NPs	SS brackets	Thermal evaporation	[367] Zeidan et al. (2022)
Ag-NPs	Metallic brackets	Laser ablation	[413] Tawakal et al. (2023)
Ag-NPs	SS bands	Electrostatic spray-assisted vapor deposition	[364] Bahrami et al. (2023)
Ag-NPs	SS archwires	Electrochemical deposition	[368] Anand et al. (2023)
Ag-NPs + TiO_2-NPs	SS archwires	Electrochemical deposition	[368] Anand et al. (2023)
Ti	NiTi archwires	Magnetron sputtering	[375] Ozeki et al. (2003)
Ti	NiTi archwires	Sputtering	[376] Anuradha et al. (2015)
Zn	SS archwires	Thermal evaporation	[381] Karandish et al. (2021)
Metallic Oxides, Nitrides and Carbides Coatings			
TiO_2	SS archwires	Sol–gel dip-coating	[389] Chun et al. (2007)
TiO_2	NiTi archwires	Oxidation treatment	[387] Espinar et al. (2011)
TiO_2	SS brackets	Magnetron sputtering	[395] Shah et al. (2011)
TiO_2	NiTi archwires	Oxidation process	[414] Satiyorini and Pintowantoro (2013)
TiO_2	NiTi archwires	Oxidation process	[415] Pintowantoro and Setiyorini (2013)
TiO_2	SS and NiTi archwires	Sol–gel dip-coating	[390] Chhattani et al. (2014)
TiO_2	NiTi archwires	*Commercial*	[64] Krishnan et al. (2014)
TiO_2	SS archwires	Sol–gel dip-coating	[392] Özyildiz et al. (2014)
TiO_2	metallic brackets	Spin-on deposition	[399] Zhang et al. (2015)
TiO_2	SS brackets	PVD	[358] Ghasemi et al. (2017)
TiO_2	SS brackets	Magnetron sputtering	[397] Baby et al. (2017)
TiO_2	SS brackets	Magnetron sputtering	[398] Fatani et al. (2017)
TiO_2	Composite archwires	Magnetron sputtering	[409] Liu et al. (2017)
TiO_2	SS brackets	Magnetron sputtering	[407] Supriadi et al. (2019)
TiO_2	SS brackets	Magnetron sputtering	[416] Supriadi et al. (2019)
TiO_2	β-Ti and NiTi archwires	PEO	[404] Jung et al. (2019)
TiO_2	SS archwires	PVD	[403] Mollabasci et al. (2020)
TiO_2	NiTi wires	Plasma oxidation	[406] Campeol et al. (2020)
TiO_2	NiTi wires	NH_3 treatments	[417] Kurtoğlu et al. (2020)
TiO_2	SS brackets	Magnetron sputtering	[418] Math et al. (2021)
TiO_2	SS archwires	Sol–gel dip-coating	[400] Kielan-Grabowska et al. (2021)
TiO_2	SS wires	Sol–gel dip-coating	[401] Bącela et al. (2022)
N-doped TiO_2	SS brackets	Magnetron sputtering	[394] Cao et al (2013)
N-doped TiO_2	composite archwires	Magnetron sputtering	[409] Liu et al. (2017)
N-doped TiO_2	SS brackets	Magnetron sputtering	[393] Salehi et al. (2018)
$TiO_{2-x}N_y$	Metallic brackets	Magnetron sputtering	[402] Li et al. (2014)
TiO_xN_y	NiTi wires	NH_3 treatments	[417] Kurtoğlu et al. (2020)
Nano-Ag/TiO_2	metallic brackets	Spin-on deposition	[399] Zhang et al. (2015)
Ag-TiO_2	SS brackets	Magnetron sputtering	[398] Fatani et al. (2017)
TiO_2 + Ag-NPs	SS archwires	Sol–gel dip-coating	[400] Kielan-Grabowska et al. (2021)
TiO_2 + Ag-NPs	SS wires	Sol–gel dip-coating	[401] Bącela et al. (2022)
TiN	NiTi archwires	*Commercial*	[419] Kim and Johnson (1999)
TiN	SS brackets	Ion plating	[420] Kao et al. (2002)
TiN	NiTi and NiTiCu archwires	Nitrogen gas difffusion	[421] Gil et al. (2004)
TiN	NiTi wires	*Commercial*	[422] Iijima et al. (2010)
TiN	SS brackets	Ion plating	[423] Huang et al. (2010)
TiN	SS brackets	Ion plating	[424] Kao et al. (2011)
TiN	SS brackets	*Commercial*	[115] Saporeti et al. (2012)
TiN	NiTi archwires	Chemical deposition	[414] Setiyorini and Pintowantoro (2013)
TiN	NiTi archwires	*Commercial*	[339] Katić et al. (2014)
TiN	NiTi wires	*Commercial*	[332] Katić et al. (2017)
TiN	NiTi archwires	*Commercial*	[340] Katić et al. (2014)
TiN	NiTi archwires	*Commercial*	[64] Krishnan et al. (2014)
TiN	NiTi archwires	*Commercial*	[425] Rongo et al. (2014)
TiN	SS brackets	Ion plating	[426] Zuo et al. (2015)
TiN	NiTi and β-Ti archwires	*Commercial*	[427] Rongo et al. (2016)
TiN	NiTi archwires	*Commercial*	[344] Musa Trolić (2017)
TiN	NiTi archwires	*Commercial*	[341] Katić et al. (2018)
TiN	SS and NiTi archwires	Ion plating	[428] Sugisawa et al. (2018)
TiN	NiTi archwires	*Commercial*	[345] Trolic et al. (2019)
TiN	NiTi archwires	*Commercial*	[279] Mlinaric et al. (2019)
TiN	NiTi archwires	*Commercial*	[346] Musa Trolic et al. (2019)
TiN	NiTi archwires	NH_3 treatments	[417] Kurtoğlu et al. (2020)
TiN	SS brackets and wires; NiTi archwires	Magnetron sputtering	[429] Arici et al. (2021)

Table 9. *Cont.*

Coatings	Substrate Materials	Deposition Methods	Literature References
TiN	SS brackets	Cathodic cage	[430] Teixeira et al. (2021)
TiN	NiTi archwires	*Commercial*	[338] Osmani et al. (2022)
TiN	SS archwires	Ion plating	[49] Ito et al. (2022)
Ti/TiN	NiTi archwires	Magnetron sputtering	[431] Liu et al. (2014)
TiAlN	β-Ti archwires	Cathodic arc PVD	[432] Krishnan et al. (2011)
TiAlN	β-Ti archwires	Cathodic arc PVD	[433] Krishnan et al. (2012)
TiN doped with CaP	SS brackets	Cathodic cage	[430] Teixeira et al. (2021)
ZrO_2	SS, NiTi and β-Ti archwires	Sol–gel	[434] Golshah and Feyli (2022)
Al_2O_3	SS brackets and wires; NiTi archwires	Magnetron sputtering	[429] Arici et al. (2021)
Al-SiO_2	NiTi archwires	Magnetron sputtering	[435] Wu et al. (2022)
Black oxide	NiTi archwires	*Commercial*	[436] Krishnan et al. (2012)
ZnO	SS brackets	Magnetron sputtering	[418] Math et al. (2021)
CrN	SS brackets and wires; NiTi archwires	Magnetron sputtering	[429] Arici et al. (2021)
CrC	SS archwires	Electroplating	[348] Usui et al. (2018)
WC/C	β-Ti archwires	Magnetron sputtering	[432] Krishnan et al. (2011)
WC/C	β-Ti archwires	Magnetron sputtering	[433] Krishnan et al. (2012)
Metal Oxide-Based NPs coatings			
ZnO	SS brackets	Spray pyrolysis	[437] Ramazanzadeh et al. (2015)
ZnO-NPs	SS wires	Chemical solution method	[438] Kachoei et al. (2015)
ZnO	SS archwires	Bath immersion	[439] Behroozian et al. (2016)
ZnO	NiTi archwires	Chemical deposition	[440] Kachoei et al. (2016)
ZnO	NiTi archwires	Electrochemical deposition	[441] Hammad et al. (2020)
ZnO	NiTi archwires	Chemical precipitationCVDSol–gel method	[442] Gholami et al. (2021)
ZnO	SS brackets and archwires	Sol–gel method	[443] Elhelbawy and Ellaithy (2021)
ZnO-NPs	SS bracktes	Thermal evaporation	[367] Zeidan et al. (2022)
ZnO-NPs	NiTi archwires	Hydrothermal method	[444] Palanivel et al. (2022)
ZnO-NPs	SS archwires	Chemical precipitationHydrothermal method	[445] Tanbakuchi et al. (2022)
AlO-NPs	NiTi archwires	Hydrothermal method	[444] Palanivel et al. (2022)
CuO	SS brackets	Spray pyrolysis	[437] Ramazanzadeh et al. (2015)
CuO-NPs	SS brackets	Dip-coating	[446] Ameli et al. (2022)
CuO-NPs	SS brackets	Dip-coating	[447] Ameli et al. (2022)
ZnO-CuO	SS brackets	Spray pyrolysis	[437] Ramazanzadeh et al. (2015)
Ag-NPs+ZnO-NPs	SS brackets	Thermal evaporation	[367] Zeidan et al. (2022)
Ag-HA-NPs	SS brackets	Dip-coating	[446] Ameli et al. (2022)
Ag-HA-NPs	SS brackets	Dip-coating	[447] Ameli et al. (2022)
TiO_2-NPs	NiTi archwires	Magnetron sputtering	[448] Venkatesan et al. (2020)
TiO_2-NPs	SS brackets	Dip-coating	[446] Ameli et al. (2022)
TiO_2-NPs	SS brackets	Dip-coating	[447] Ameli et al. (2022)
TiO_2-NPs	SS archwires	Dip-coating	[449] Silveira et al. (2022)
TiO_2-NPs	SS archwires	Magnetron sputtering	[368] Anand et al. (2023)
TiO_2-NPs	β-Ti and NiTi archwires	Dip-coating	[450] Chaturvedi et al. (2023)
IF-NPs-reinforced Metal-based Coatings			
WS_2-reinforced Ni	SS archwires	Electrochemical co-deposition	[451] Redlich et al. (2008)
WS_2-reinforced Ni	SS archwires	Electrochemical co-deposition	[452] Samorodnitzky-Naveh et al. (2010)
WS_2-reinforced Ni-P	SS archwires	Electroless deposition	[453] Katz et al. (2006)
WS_2-reinforced Ni-P	SS archwires	Electroless deposition	[454] Redlich et al. (2008)
MoS_2-reinforced Ni	SS archwires	Electrochemical co-deposition	[455] Gracco et al. (2019)
WS_2-reinforced Co	NiTi archwires	Co-electrodeposition	[456] Samorodnitzky-Naveh et al. (2009)
WS_2-reinforced Co	NiTi archwires	Co-electrodeposition	[452] Samorodnitzky-Naveh et al. (2010)

- Ti–N System:

The titanium nitride system is commercially accessible as TiN-coated brackets and archwires [64,115,279,332,338,340,341,344–346]. The typically gold-colored TiN coatings may be produced onto orthodontic-compatible metals by several methods, including pulsed high-energy density plasma treatment with Ti electrodes [457], nitrogen gas (N_2) diffusion [421] and ammonia gas (NH_3) treatments [417], plasma immersion ion implantation [422,458], ion beam assisted deposition [459], ion plating [420,423,424,426,428,460], and other PVD

methods [396,429,431,461]. As presented below, there is no academic consensus on the corrosion susceptibility of these nitride coatings in orthodontics.

Kao et al. [420] attempted to reduce the corrosion of SS brackets in artificial saliva by TiN ion plating, but no statistically significant difference between coated and uncoated brackets was observed on both metallic ion release and biocompatibility. Later attempts also failed: TiN ion-plated metallic brackets were unable to reduce friction against archwires in several media [423,424]. Opposite results by Zuo et al. [426] showed super low coefficients of friction (CoF < 0.03) under humid wear conditions in artificial saliva, high hardness (14.62 GPa), and improved adhesion of coated SS316L disks and brackets. Such a combination of mechanical and tribological properties is expected to decrease wear and release of toxic elements during orthodontic treatments. In addition, the coated samples presented an appealing golden color.

Jin et al. [460] were also optimistic about their research work: TiN ion-plated NiTi substrates may lead to improved biocompatibility, as fibroblasts' adhesion and proliferation increased, whereas no cracks were reported during the three-point bending test after a 4099 N load. Deposition of the coatings onto SS and NiTi archwires diminished corrosion susceptibility in artificial saliva and friction force against metallic brackets up to a bracket/archwire angulation of 10°, as reported by Saporeti and coauthors [428]. The resulting higher tensile strength and stiffness of the SS wires are particularly useful properties in orthodontics. In turn, Mlinaric et al. [279] argued that, depending on the oral antiseptic used, the mechanical behavior of nitride NiTi (elastic properties and force delivery) can be affected, and Ni release may have been promoted during immersion in both the artificial saliva and the antiseptic.

A single TiN layer and a TiN/TiO_2 multilayer deposited onto AISI 316L substrates by reactive magnetron sputtering effectively reduced the adhesion of calcium-precipitating bacteria, which are responsible for the formation of dental calculus [396]. Wang et al. [462] previously nitrided the surface of commercially pure Ti—a duplex treatment—decreasing ion release by almost 5 times compared to untreated samples when exposed to F-containing artificial saliva. When single Ti was used as a 200 nm thick interlayer for a 1 μm TiN coating on NiTi orthodontic archwires, potentiodynamic tests showed that localized corrosion was prevented in acidulated artificial saliva, both under loading and unloading conditions [431]. Other researchers reported that 1.6 and 3.2 μm thick Ti/TiN multilayer systems (16 bilayers) were more effective in improving the corrosion resistance of 316L SS samples than a simple TiN monolayer. This was demonstrated by using NaCl and Hank's solutions [463]. However, the hardness decreased from ~13 to 10 GPa when using the multilayer system due to the presence of the softer Ti adhesion layers [463].

In opposition to the Ti–O system, no Ag addition to TiN coatings was found in the literature (Table 9). Instead, research work concerning the aluminum (Al) effect on TiN coatings by cathodic arc PVD was carried out [432,433]. The presence of Al (content not specified) decreased through-coating defects and prevented corrosion of the β-Ti archwires but maintained the mechanical properties (namely under unloading process). The results indicated that the TiAlN films effectively protected the wire during potentiodynamic tests in F-containing artificial saliva (no ionic release or coating damage was detected), while offering good biocompatibility without inducing apoptosis of oral epithelial cells [432].

Teixeira et al. [430] attempted to improve the surface properties of SS brackets by depositing single and calcium phosphate (CaPa)-containing TiN coatings via the cathodic cage method. Unfortunately, these coatings were unable to prevent the formation of *S. mutans* biofilms on the surface [430].

Recently, Kurtoğlu et al. [417] presented a simple and low-cost NH_3 surface treatment in He atmosphere at 700 °C and assessed the in vitro corrosion resistance against artificial saliva. Thin TiN and TiO_xN_y films (<1 μm thick) significantly reduced the Ni release from shape-memory NiTi substrates. Nonetheless, the high treatment temperature used may have negatively impacted the mechanical properties of the bulk orthodontic alloys [357,464].

- Zr–O and Al–O Systems:

Both Zr–O and Zr–Al systems—zirconia (ZrO_2) and alumina (Al_2O_3)—are well recognized as bulk biomaterials due to their attractive combination of high corrosion resistance, low friction, high strength, and high wear resistance [465,466]. As surface material, ZrO_2 coatings (1 µm thick) have been deposited onto AISI 316L substrates by electron-beam PVD [467]. These coatings showed excellent corrosion resistance in NaF-containing Fusayama–Meyer artificial saliva (2% NaF, pH = 3.0), while both *S. mutans* bacterial adhesion and fibroblast cytotoxicity decreased in vitro [467]. More recently, Golshah and Feyli [434] attempted to deposit ZrO_2 on SS, TMA, and NiTi archwires using the sol–gel technique. Unfortunately, the researchers only successfully synthesized the coating on the TMA archwires, without a significant decrease in the static or kinetic friction against SS brackets [434].

Arici et al. [429] studied the binary Al–O system, deposited by magnetron sputtering, on SS brackets and SS and NiTi archwires. For this investigation, the researchers [429] designed a special experimental procedure to simulate the intraoral temperature variation (involving 250 thermal cycles, from −5 to +55 °C) and mechanical toothbrushing with fluoride toothpaste (12 h). The Al_2O_3 coatings presented good thermal, frictional, and brushing behavior. The referred work [429] also intended to study other sputtered coatings, TiN and CrN, which presented worse results in similar test conditions. While the TiN coatings showed small peeling areas, the CrN ones exhibited large detachment areas from the substrate. In fact, the CrN coatings failed the designed thermal, frictional, and brushing tests. The researchers therefore concluded that these Cr-based coatings were unsuitable for decreasing the coefficient of friction (CoF) in orthodontics. The best wear behavior was found for Al_2O_3 (or TiN)-coated archwires against metal brackets (CoF = 0.207 and 0.372, respectively). Wu et al. [435] deposited Al–SiO_2 coatings by magnetron sputtering that smoothed the surface of NiTi and SS archwires and enhanced their overall corrosion resistance with no apparent cytotoxicity.

- Zn–O System:

The Zn–O system, zinc oxide (ZnO), was also deposited as a coating by magnetron sputtering on SS brackets by Math et al. [418]. The coatings underwent thermal oxidation at 500 °C in an open-air furnace for 5 h. The researchers studied both anti-adhesion and antibacterial properties against *S. mutans*. The reported results were disappointing because they showed that ZnO coatings were unable to inhibit adhesion and growth of that bacterial strain.

- W–C, W–N, Cr–C, and Cr–N Systems:

The binary W–C, W–N, Cr–C, and Cr–N systems—included in the well-known protective hard coatings for high-speed cutting operations [385]—have also merited attention in orthodontics. β-Ti archwires were functionalized with sputtered tungsten carbide/carbon (WC/C) coatings by Krishnan et al. [432,433]. The idea was to achieve protection against fluoride-induced corrosion. It was concluded that these coatings were mechanically stable and biocompatible; however, to the identified surface defects could act as initiators of pitting corrosion. In fact, deeper and accentuated cracks were observed in the WC/C coating after fluoride immersion tests [432]. The approach by single W and W–N [468] sputtered coatings was also disappointing on AISI 316L flat samples immersed in artificial saliva immersion. The external passive oxide phase revealed unprotective behavior, accompanied by severe and deep crack formation, as shown in Figure 18.

Usui et al. [348] compared the mechanical, frictional, and aesthetic properties of hard Cr–C-plated SS wires with uncoated, Rh-, and polymeric-coated SS orthodontic archwires. While the three-point bending test revealed similar values of flexural strength and modulus among all wire types, frictional forces significantly decreased against ceramic brackets. Compared with uncoated, Rh-, and polymer-coated samples, both maximum static and kinetic, dry, and wet frictional forces decreased by approximately 15–18, 42–45, and 17–22%, respectively.

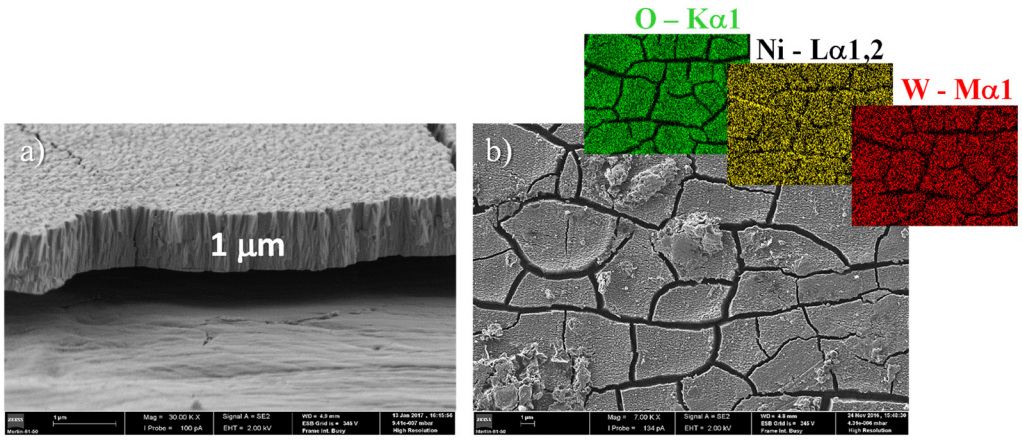

Figure 18. SEM micrographs of as-deposited W–N sputtered coatings (**a**) and its surface morphology after (**b**) Fusayama–Meyer solution aging (37 °C, 30 days, pH 8.3) coupled with EDS elemental maps distribution (adapted from [468], own work).

5.1.3. Metal Oxide-Based Nanoparticles Coatings

Metal oxide-based NPs have been used to improve the performance of biocomponent surfaces, particularly as antimicrobial agents for multiple biomedical applications [469]. According to the literature, the following types of metallic oxide NPs have been considered to cover metallic fixed appliances: ZnO- [367,437–443,445] and/or CuO-NPs [437,446,447], AlO-NPs [444], TiO_2-NPs [446–449], and Ag–HA-NPs [446,447], as presented in Table 9.

ZnO-NPs are novel dental materials with several potential applications in dentistry, including prosthodontic, endodontic, restorative, implantology, periodontal, and orthodontic fields [470]. As protective coatings, the research works concerning ZnO-NPs intend to reduce not only the bacterial adhesion and activity [367,437,440–442], but also the bracket/wire contact wear [439–441,443–445].

Excellent antibacterial results of 93, 96, and 98% against *S. mutans* have been achieved by Gholami et al. [442] with ZnO-NPs deposited by CVD, chemical precipitation and sol–gel methods, respectively, on the surface of NiTi archwires. Hammad et al. [441] also described excellent antibacterial activity for electrochemically deposited ZnO-NPs onto NiTi archwires against different bacterial strains (*S. aureus*, *S. pyogens* and *E. coli*). Other researchers [437] support the antibacterial effect of ZnO-NPs deposited by spray pyrolysis on SS brackets and by a chemical method on NiTi archwires [439]. Nonetheless, a mixture of CuO–ZnO–NPs seems to be more effective than a single ZnO-NPs for SS brackets, by using the spray pyrolysis method. Yet, the color of the surface unsuitably changed to copper (orange-red) [437].

Zeidan et al. [367] compared the antibacterial effect of ZnO- and/or Ag-NPs deposited on SS brackets by thermal evaporation. The researchers found the highest antibacterial effect against *S. mutans* and *L. acidophilus* for the ZnO/Ag-NPs coating. Interestingly, all coatings showed antibacterial effects that persisted for up to 3 months [367].

Concerning the mechanical properties, Elhelbawy and Ellaithy [443] deposited ZnO-NPs coatings via sol–gel on SS brackets and/or archwires, allowing a 64% decrease in the mean friction forces.Other researchers reported more modest reductions for coated NiTi archwires against uncoated SS brackets: ~34% [441,444], ~21% [440] (bracket/wire angulation up to 10°), and ~11% [445]. Palanivel et al. [444] also compared the friction forces of SS brackets against uncoated, ZnO-NPs-, or AlO-NPs-coated NiTi archwires, finding that the latter coating type showed an intermediate performance. ZnO-NPs-coated SS bands showed in vitro biocompatibility with HGF cells [364].

A different approach for reducing bacterial adhesion and subsequent enamel damage consisted of magnetron-sputtered TiO_2 nanoparticles (TiO_2-NPs) [448]. A thin layer (~81 nm) successfully prevented in vivo bacterial adhesion to NiTi archwires for up to 1 month. Coating physical stability was the main downside: Around 60% of the coating

showed adhesion failures [448]. Ameli et al. [446,447] showed that TiO_2-NPs and Cu-NPs coatings on SS brackets deposited by a sol–gel method possessed antibacterial properties and could significantly reduce the friction against metallic archwires. Chaturvedi and colleagues [450] dip-coated β-Ti and NiTi archwires with TiO_2-NPs using different concentrations and dipping durations. The authors found an optimum NP concentration range of 1:2–1:6 for 48 h and a decrease in frictional forces for almost all coated wires [450].

5.1.4. Inorganic Fullerene-like Nanoparticles (IF-NPs)-Reinforced Metal-Based Coatings

Metal-based coatings containing inorganic fullerene-like nanoparticles (IF-NPs) are also listed in this review (Table 9). Round-shaped IF-NPS have been embedded into a metallic matrix to achieve composite coatings with self-lubricating properties. The wear mechanism is particularly relevant in bracket/archwires contacts in the salivary environment. Tungsten disulfide (WS_2) was the IF-NPs of choice for nickel (Ni) [451,452,455], nickel–phosphorus (Ni–P) [453,454] and cobalt (Co) [452,456] matrixes, probably due to its excellent dry bulk lubricity.

Samorodnitzky-Naveh et al. [456] reinforced Co coatings with WS_2 IF-NPs by using co-electrodeposition onto NiTi alloys. Successfully, the friction coefficient of the NiTi plates decreased by 66%—from 0.26 to 0.09 (as evaluated by pin-on-disk). Moreover, friction tests of the coated NiTi wires against SS brackets revealed minimum static and kinetic CoF of 0.080 and 0.061, respectively, for a maximum bracket/wire angulation of 5° [456].

Redlich and colleagues [451,453,454] selected Ni and Ni–P matrixes to study the WS_2 IF-NPs effect over SS archwires. Reduced friction forces were claimed, both under dry and wet conditions: 17 and 54% decrease for 0 and 10° bracket/wire angulation, respectively. The 3–5 μm thick Ni-P/[Ni-P + IF-NPs] coatings reduced CoF by half (to ~0.05) after a 50-cycle ball-on-flat test [454].

Recently, Gracco et al. [455] developed and compared molybdenum disulfide (MoS_2)—another solid lubricant—with WS_2 IF-NPs-containing Ni coatings (~20 and 15 μm thick, respectively) on SS orthodontic wires. The coatings were synthesized by electrochemical co-deposition. Overall, both coating types always decreased the in vitro frictional forces against two types of metallic brackets, under dry and wet conditions, for 0 and 5° bracket/wire angulation. The researchers [455] observed no significant damage for minimum bending of the composite Ni/IF-NPs-coated wires, but a complete failure was registered for a maximum bending of 5° angulation.

The use of IF-NPs-containing coatings is, therefore, promising, but further research on their behavior under simulated oral conditions is still required, mainly regarding the effect of sulphur (S) on surfaces' biocompatibility. Based on the reasons mentioned in the previous chapter, the selection of Ni-based matrices is quite undesirable in orthodontics.

Despite the large number of scientific works, further studies on durability, corrosion and wear resistance, and possible cytotoxicity to oral epithelial cells are still required for metals and their oxide, nitride, or carbide phase coatings and thin films.

5.2. Polymer-Based Coatings

Several polymeric coatings are clinically available for fixed orthodontics (Table 10). Polytetrafluoroethylene (PTFE) is, undoubtedly, the most studied thermoplastic polymer for orthodontic applications, followed by epoxy resins. Recently, research on polymeric composite materials has also gained importance, particularly those reinforced by ceramic NPs.

5.2.1. Thermoplastic and Thermoset Coatings

- Polytetrafluoroethylene (PTFE):

Polytetrafluoroethylene, also known as Teflon™, is widely used in multiple fields, including many medical applications [471,472] for its outstanding biocompatibility and inertness. In orthodontics, PTFE aesthetic coatings for archwires are commercially available due to their suitable white color, similar to the tooth's tone.

A recent study [473] reported that PTFE coatings onto SS, NiTi, and β-Ti wires "*exhibited low coloration, low microbial adhesion, low friction against metallic brackets, and high tolerance of detachment and wear against toothbrushing*", especially if produced at a low temperature (200 °C) using a two-stage spraying technique. Other researchers agree that PTFE coatings onto metallic archwires are highly stable; significantly improve the corrosion resistance of the substrates in acidulated artificial saliva [330,474,475], in Ringer's solution [64], and in contact with certain food products [476,477]; maintain the surface morphology after cyclic mechanical loading of coated wires [330]; and decrease frictional forces against brackets [478,479]. PTFE coatings can also reduce biofilm adhesion to SS brackets. An in situ study [480] revealed that biofilm accumulation covered more than 22% of the uncoated brackets, in contrast to scarcely 4% of the PTFE-coated brackets. Still, the coated areas under high shear forces (e.g., bracket wings) partially degraded, exposing the metallic substrate.

Other reports, however, concluded that PTFE coatings directly affect load-deflection properties [333,481,482], surface roughness [333,481,483,484] and hardness [335] of NiTi archwires; undergo slight discoloration after 3 to 4 weeks of immersion in a staining solution [335,485]; and suffer from significant to severe coating detachment, both in vitro [486,487] and in vivo [373,488–490] (Figure 19). For instance, Rongo et al. [427] concluded that Teflon^{TM}-coated NiTi archwires had similar biocompatibility to uncoated ones. Nevertheless, the mechanical response was different: Friction forces against metallic and aesthetic brackets increased, besides suffering delamination during 1 month of clinical use [425]. Scratch tests performed by Silva et al. [491] with four commercially available archwires coated with a polymer mixture (polytetrafluoroethylene and polyester) revealed high elasticity recoveries of more than 60%, but this mechanical behavior was accompanied by "*delamination, crack propagation, and debris generation along the coatings scratches*". Lin et al. [492] ascertained that prolonged water immersion (up to 4 weeks) of PTFE-coated archwires had negatively impacted the resistance to sliding against ceramic brackets. Thus, the functionality and durability of such coatings in the oral environment were questioned.

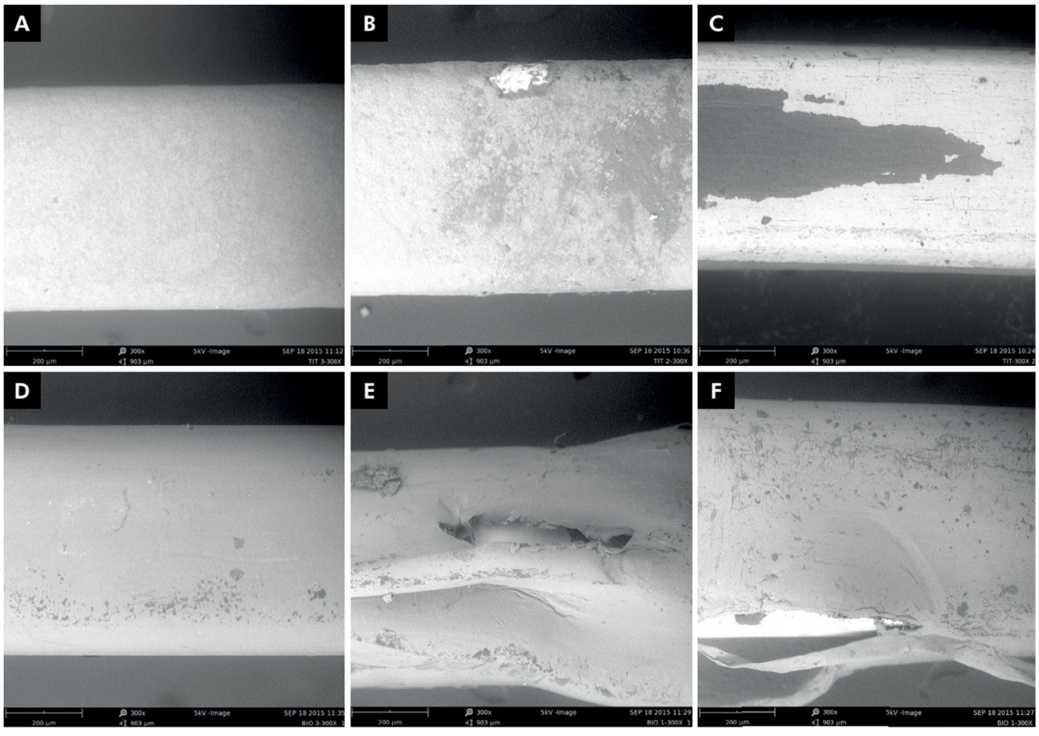

Figure 19. Photomicrographs of coated wires: (**A**) as-received Titanol® Cosmetic, (**B**,**C**) post clinical Titanol® Cosmetic, (**D**) as-received Biocosmetic, (**E**,**F**) post clinical Biocosmetic (reproduced from [488]).

- Epoxy Resins:

Epoxy resins (containing an epoxide group) are another widely used polymer in medicine [493], existing as commercially available aesthetic coatings for archwires with an appealing white color.

Some researchers concluded that epoxy-coated archwires reduced bacterial adhesion [63], prevented dental plaque accumulation [494], and possessed similar mechanical properties (e.g., unloading force) [336] and surface roughness [495] to uncoated wires. Back in 1999, Kim and Johnson [419] recommended the use of epoxy-coated NiTi archwires due to their superior corrosion resistance when compared with uncoated ones. Similarly, Amorim et al. [342] concluded that epoxy resin coatings effectively enhanced the corrosion resistance of NiTi archwires in artificial saliva, for instance, by decreasing Ni release. Nevertheless, several in vitro studies reported major flaws, namely: low coating stability, durability, and color stability [138,348,474,485,486,496,497], lower hardness [335], and loading/unloading forces [497,498], or increased roughness [333,412,481] and friction forces against brackets [499], which may limit the clinical performance.

Abdulkader et al. [474] performed a simple durability test: Coated and uncoated archwires were immersed in artificial saliva at 3.5 and 6.75 pH values for 28 days at 37 °C. After the immersion period, samples were washed with normal saline solution, fixed to brackets with elastomeric ligatures, and subjected to mechanical stress by toothbrushing (210 s). The results revealed "*rupture, roughness, and coatings damage in multiple locations*". Under acidic conditions, 48% coating loss was reported (Figure 20), while for more neutral pH (6.75), this value decreased to 31%. Alavi and Hosseini [496] immersed coated and uncoated samples in a commercial artificial saliva (at pH 6.7 and 37 °C), refreshed every day. After 3 weeks, they performed 500 thermocycles—10 min at 5 °C, 10 s at room temperature, and 10 min at 55 °C—and then a three-aesthetic bracket bending test. Very recently, Aboalnaga and colleagues [500] tested three commercially available epoxy-coated NiTi archwires and found undesirable surface changes after a one-month immersion test in artificial saliva, including lower surface hardness and higher roughness values.

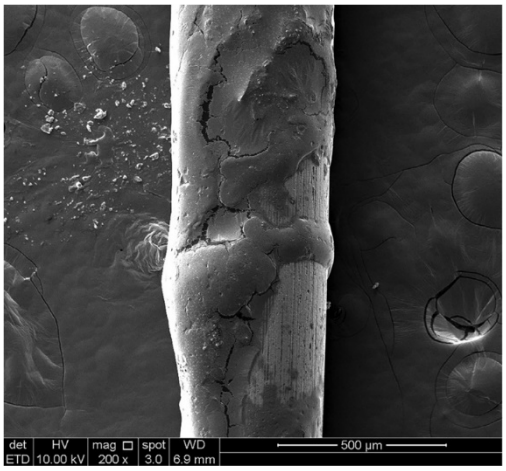

Figure 20. Epoxy coating loss on an archwire when exposed to acidic artificial saliva (reprinted from [474], Copyright (2020), with permission from Elsevier).

Elyayyan et al. [498] obtained lower loading and unloading forces of new coated wires when compared with uncoated wires in a three-point bending test. The same researchers [501] also argued that epoxy-coated NiTi wires "*had low aesthetic value*", as "*25 % of the coatings was lost within 33 days* in vivo *and surface morphology showed severe deterioration*". Abdulkhabeer et al. [489] found mean coating losses between 18.3 and 28.6% of epoxy-coated NiTi archwires after 8 weeks of orthodontic treatment. The color stability of these coatings has also been questioned, since an extremely marked change in color was noticed in a 21-day immersion test in coffee staining solutions [350,485].

Shao et al. [502] used a mixture of epoxy resin and PTFE suspension, in different proportions and with white and yellow dies, to dip-coat NiTi archwires. Coatings with the color of human teeth were obtained. The results from the acute toxicity and mucous membrane irritation tests showed no negative signs regarding in vivo biocompatibility [502].

- Polyether-ether-ketone (PEEK):

Polyether-ether-ketone resin is a cheap and non-cytotoxic advanced polymer that meets several chemical, tribological, and mechanical properties for orthodontics. To overcome the low PEEK adhesion to NiTi archwires, Sheiko et al. [503] proposed a new method to increase the adhesion of the polymeric coating. After cleaning the Nitinol archwires, they performed electrochemical polishing to create a thin and smooth TiO_2 interlayer, above which a homogeneous PEEK top layer (~12 μm) grew via dip-coating. The coatings strongly adhered to the metallic substrate without delamination after immersion in Hank's solution for up to 31 days, or 7 million compressing/stretching cycles at 20% strain. Regarding biocompatibility and corrosion properties, the coated archwires showed negligible Ni release during the immersion test and no cytotoxic effect on murine fibroblasts. However, local pressures above 2 GPa may disrupt the PEEK coating and unprotect the NiTi alloy surfaces against corrosion reactions [503].

More recently, Shirakawa et al. [504] followed a different method to cover NiTi archwires with PEEK resin: A simple outer polymeric tube around the arches. Friction tests revealed lower friction forces against brackets when compared to uncoated SS-, CoCr, and NiTi archwires. Since coated wires were able to slide more easily in contact, bracket slots maintained the as-received surface conditions [504].

Table 10. Overview of polymer-based coatings deposited on different orthodontic appliances and substrates. PECVD: plasma-enhanced chemical vapor deposition; n.s.—not specified.

Coatings	Substrates	Deposition Method	Literature References
Thermoplastic and Thermoset Coatings			
PTFE	NiTi and SS archwires	*Commercial*	[330] Neumann et al. (2002)
PTFE	NiTi and SS archwires	*Commercial*	[479] Husman et al. (2002)
PTFE	SS brackets	*n.s.*	[480] Demling et al. (2010)
PTFE	SS and NiTi archwires	*Commercial*	[478] Farronato et al. (2012)
PTFE	NiTi archwire	*Commercial*	[490] Zegan et al. (2012)
PTFE	NiTi archwire	*Commercial*	[64] Krishnan et al. (2014)
PTFE	NiTi archwires	*Commercial*	[425] Rongo et al. (2014)
PTFE	NiTi archwires	*Commercial*	[475] Mareci et al. (2015)
PTFE	NiTi archwires	*Commercial*	[481] Ryu et al. (2015)
PTFE	NiTi archwires	*Commercial*	[484] Choi et al. (2015)
PTFE	NiTi archwires	*Commercial*	[476] Earar et al. (2016)
PTFE	NiTi archwires	*Commercial*	[427] Rongo et al. (2016)
PTFE	NiTi archwires	*Commercial*	[333] Albuquerque et al. (2017)
PTFE	NiTi archwires	*Commercial*	[505] Rego et al. (2017)
PTFE	NiTi archwires	*Commercial*	[477] Matei et al. (2016)
PTFE	NiTi archwires	*Commercial*	[373] Shirakawa et al. (2017)
PTFE	NiTi archwires	*Commercial*	[485] Rego et al. (2017)
PTFE	NiTi archwires	*Commercial*	[310] Matias et al. (2018)
PTFE	NiTi archwires	*Commercial*	[411] Asiry et al. (2018)
PTFE	NiTi archwires	*Commercial*	[483] Dokku et al. (2018)
PTFE	SS archwires	*Commercial*	[487] Shahabi et al. (2018)

Table 10. *Cont.*

Coatings	Substrates	Deposition Method	Literature References
PTFE	NiTi archwires	*Commercial*	[335] Alsanea and Shehri (2019)
PTFE	NiTi archwires	*Commercial*	[347] Costa Lima (2019)
PTFE	SS, NiTi and β-Ti archwires	Thermal spraying	[473] Kameda et al. (2020)
PTFE	NiTi archwires	*Commercial*	[474] Abdulkader et al. (2020)
PTFE	NiTi archwires	*Commercial*	[489] Abdulkhabeer et al. (2020)
PTFE	NiTi archwires	*Commercial*	[486] Jejurikar et al. (2020)
PTFE	NiTi archwires	*Commercial*	[337] Batista et al. (2020)
PTFE	NiTi archwires	*Commercial*	[482] Elsaka et al. (2021)
PTFE	SS archwires	*Commercial*	[492] Lin et al. (2021)
PTFE	β-Ti and SS archwires	Spray treatment	[506] Zhou et al. (2023)
Epoxy	NiTi arcwhires	*Commercial*	[419] Kim and Johnson (1999)
Epoxy	NiTi archwires	*Commercial*	[501] Elayyan et al. (2008)
Epoxy	NiTi archwires	*Commercial*	[498] Elayyan et al. (2010)
Epoxy	NiTi archwires	Electrostatic powder deposition	[138] Bandeira et al. (2011)
Epoxy	NiTi archwires	*Commercial*	[496] Alavi et al. (2012)
Epoxy	NiTi archwires	*Commercial*	[494] Raji et al. (2014)
Epoxy	NiTi archwires	*Commercial*	[64] Krishnan et al. (2014)
Epoxy	NiTi archwires	*Commercial*	[63] Kim et al. (2014)
Epoxy	NiTi archwires	*Commercial*	[497] Pop et al. (2015)
Epoxy	NiTi archwires	*Commercial*	[484] Choi et al. (2015)
Epoxy	NiTi archwires	*Commercial*	[333] Albuquerque et al. (2017)
Epoxy	NiTi archwires	*Commercial*	[505] Rego et al. (2017)
Epoxy	SS archwires	*Commercial*	[374] Usui et al. (2017)
Epoxy	NiTi archwires	*Commercial*	[485] Rego et al. (2017)
Epoxy	SS archwires	*Commercial*	[348] Usui et al. (2018)
Epoxy	NiTi archwires	*Commercial*	[310] Matias et al. (2018)
Epoxy	NiTi archwires	*Commercial*	[411] Asiry et al. (2018)
Epoxy	NiTi archwires	*Commercial*	[483] Dokku et al. (2018)
Epoxy	NiTi archwires	*Commercial*	[335] Alsanea and Shehri (2019)
Epoxy	NiTi archwires	*Commercial*	[499] Dragomirescu et al. (2019)
Epoxy	NiTi archwires	*Commercial*	[495] Shamohammadi et al. (2019)
Epoxy	NiTi archwires	*Commercial*	[474] Abdulkader et al. (2020)
Epoxy	NiTi archwires	*Commercial*	[336] Pinzan-Vercelino et al. (2020)
Epoxy	NiTi archwires	*Commercial*	[489] Abdulkhabeer et al. (2020)
Epoxy	NiTi archwires	*Commercial*	[486] Jejurikar et al. (2020)
Epoxy	NiTi archwires	*Commercial*	[350] Ramasamy et al. (2020)
Epoxy	NiTi archwires	*Commercial*	[412] Madasamy et al. (2021)
Epoxy	SS archwires	*Commercial*	[492] Lin et al. (2021)
Epoxy	NiTi archwires	*Commercial*	[342] Amorim et al. (2022)
Epoxy	β-Ti and SS archwires	Spray treatment	[506] Zhou et al. (2023)
Epoxy	NiTi archwires	*Commercial*	[500] Aboalnaga et al. (2023)
PEEK	Nitinol wires	Dip-coating deposition	[503] Sheiko et al. (2016)
PEEK tubes	SS, NiTi, and CoCr archwires	Tube coverage	[504] Shirakawa et al. (2018)
PE	NiTi archwires	*Commercial*	[330] Neumann et al. (2002)

Table 10. *Cont.*

Coatings	Substrates	Deposition Method	Literature References
PE	NiTi archwires	*Commercial*	[479] Husman et al. (2002)
PEN (PE naphthalate)	SS archwires	*Commercial*	[61] Ito et al. (2022)
Polyamide	NiTi archwires	Dipping treatment	[507] Bravo et al. (2014)
Epoxy + PTFE	NiTi archwires	Dip-coating deposition	[502] Shao et al. (2009)
PTFE + polyester	SS and NiTi archwires	*Commercial*	[491] da Silva et al. (2015)
Other Polymer Coatings			
Lysozyme	Composite archwires	Coating protein deposition	[318] He et al. (2020)
Hexamethyldisiloxane (HMDSO)	SS brackets	PECVD	[508] Tupinambá et al. (2017)
Organosilane	SS brackets	Sol–gel method	[509] Oliveria et al. (2015)
2-methacryloyloxyethyl phosphorylcholine	SS archwires	Chemical deposition	[510] Kunimatsu et al. (2022)
Chitosan	Brackets *(n.s.)*	Freeze-drying	[511] Want et al. (2023)
Chitosan nanoparticles	SS brackets and archwires	Sol–gel method	[443] Elhelbawy and Ellaithy (2021)
Ag–chitosan nanoparticles	Metallic brackets	Laser ablation	[413] Tawakal et al. (2023)
Polyoxazoline + tryptophan	SS brackets	Plasma polymerization/Immobilization	[512] Kumarasinghe et al. (2021)
1H,1H,2H,2H-perfluorodecyltrimethoxysilane(FAS) + bovine serum albumin (BSA)	SS brackets and archwires	Chemical deposition	[513] Liu et al. (2018)
PVA hydrogel	SS archwires	Chemical deposition	[514] MingWen et al. (2023)
Polydopamine + honokiol C-dots	SS brackets	Hydrothermal method/chemical deposition	[515] Wang et al. (2023)
Butyl-3-methylimidazolium chloride	NiTi archwires	Ionic liquid coating	[516] Ahmed et al. (2021)
Parylene	SS archwires	*Commercial*	[492] Lin et al. (2021)
Parylene with Ag–Pt layer	NiTi archwires	*Commercial*	[334] Iijima et al. (2012)
Parylene with Ag–Pt layer	NiTi archwires	*Commercial*	[484] Choi et al. (2015)
Ag/biopolymer bilayer	NiTi archwires	*Commercial*	[63] Kim et al. (2014)
Ag/polymer bilayer	NiTi archwires	*Commercial*	[482] Elsaka et al. (2021)
Silicone	β-Ti and SS archwires	Dip coating	[506] Zhou et al. (2023)
Ceramic-reinforced epoxy composite	β-Ti and SS archwires	Spray deposition	[506] Zhou et al. (2023)
Polymer (?)	NiTi archwires	*Commercial*	[496] Alavi et al. (2012)
Polymer (?)	NiTi archwires	*Commercial*	[425] Rongo et al. (2014)
Polymer (?)	NiTi archwires	*Commercial*	[427] Rongo et al. (2016)
Polymer (?)	NiTi archwires	*Commercial*	[488] Argalji et al. (2017)
Polymer (?)	NiTi archwires	*Commercial*	[499] Dragomirescu et al. (2019)
Polymer (?)	NiTi archwires	*Commercial*	[495] Shamohammadi et al. (2019)
Polymer (?)	NiTi archwires	*Commercial*	[482] Elsaka et al. (2021)
NPs reinforced Polymer Coatings			
TiO_2 reinforced Epoxy	NiTi archwires	Electrophoretic deposition	[517] Xu et al. (2019)
ZnO reinforced PVP	NiTi archwires	Electrospinning	[442] Gholami et al. (2021)
ZnO reinforced PVA	NiTi archwires	Polymer composite coating	[442] Gholami et al. (2021)

- Polyethylene (PE) and Polyethylene Naphthalate (PEN):

Polyethylene is a commercially available coating for NiTi archwires [330,479]. Husman et al. [479] indicated that the plasma-based synthesis process starts by removing micro-contaminants on the wire surface through high-energy oxygen radicals, followed by the formation of titanium oxide, and finally the deposition of polyethylene. The surface is

smooth due to the filling of its depressions [330,479]. Commercial PE-coated archwires showed an apparently superior in vitro corrosion resistance than the uncoated ones, even though the rupture potential has been lowered. This may indicate different corrosion processes. Moreover, PE-coated NiTi archwires showed no changes after cyclic mechanical loading [330] and reduced the friction forces against SS brackets [479]. Ito et al. [61] recommended the use of polyethylene naphthalate—also a commercial coating—for patients with metal allergies and when wire bending is required due to the in vitro mechanical stability of coated SS archwires. Even so, only toothbrushes with soft filaments should be used for oral hygiene procedures to prevent damaging the coating and, consequently, leading to corrosion of the metallic substrate [61].

- Polyamides:

Polyamides are additional potential polymeric coatings for NiTi superelastic archwires. According to Bravo et al. [507], the dip-coated wires showed 85% less Ni release than uncoated samples after the 30-day electrochemical corrosion tests. During self-designed friction tests against SS- and Ti6Al4V-brackets, wear rates and both static and dynamic CoF were always inferior in comparison to other alloy archwires types (NiTi, TiMo, commercially pure Ti, Cu–NiTi or SS 304 AISI). In addition, both coated and uncoated wires had very similar superelastic behavior [507].

- Other Polymer Coatings:

Lysozyme coatings were used by He et al. [318] to avoid Cu corrosion from laser-welded NiTi + SS CAW. These coatings, synthesized via liquid-phase deposition, presented antimicrobial properties. *S. aureus* activity decreased from 97 down to 59%, with increased electrochemical corrosion resistance, while cytocompatibility with fibroblast cells was higher for coated samples (79 vs. 83–90%).

Hexamethyldisilane (HMDSO) films were also deposited on conventional and self-ligating SS brackets by PECVD [508]. The plasma-polymerized resulting coatings were only effective in reducing surface roughness and bacterial adhesion in conventional brackets. Their uncoated, smoother surface and more favorable external geometry led to a better film deposition than in self-ligating brackets.

Organosilane-based coatings with super-hydrophobic properties were deposited via sol–gel on SS and ceramic brackets by Oliveira and colleagues [509]. A water contact angle of ~123° was reported, in addition to an exponential decrease in in vitro biofilm accumulation with increasing water contact angle of the surface up to 24 h of incubation (Figure 21).

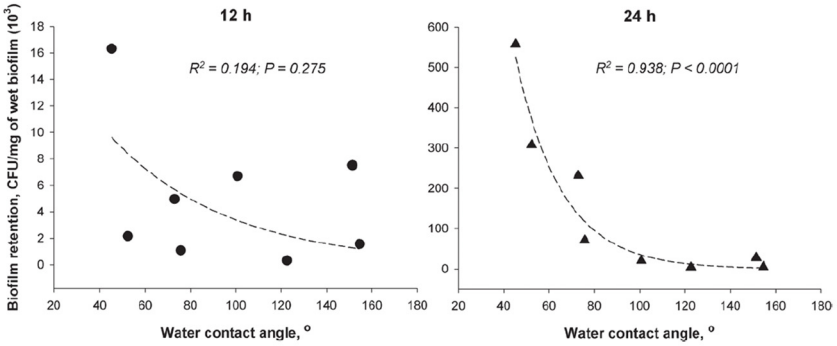

Figure 21. Biofilm retention after 12 and 24 h of incubation as a function of the water contact angle on organosilane coated brackets (used with permission of IOP Publishing, Ltd., from [509]; permission conveyed through Copyright Clearence Center, Inc.).

Chitosan—an antibacterial carbohydrate polymer—was studied as a coating for orthodontic brackets. Non-crosslinked chitosan was deposited by freeze-drying from previously dissolved chitosan in 2% (*w*/*v*) acetic acid at different concentrations (1.0, 2.5, 5.0, and

10 mg/mL). Promising results were obtained, as *S. mutans* bacterial adhesion decreased by up to 99.6% for coated brackets [511].

Chitosan-NP materials were also studied as antibacterial surfaces for orthodontics. The deposition process was performed by sol–gel thin film dip coating. A decrease in friction of SS brackets and archwires against coated and/or uncoated counterparts [443] was accessed by the researchers. A significant reduction in friction forces and *S. aureus* activity was reported as well. Ag–chitosan NPs deposited on metallic brackets showed outstanding antibacterial activity against that bacterial strain [413].

MingWen et al. [514] quite recently deposited a toughly-adherent polyvinyl alcohol (PVA) hydrogel onto SS archwires. The authors reported a remarkable decrease in friction in water (CoF as low as 0.005) and a significant decrease in *E. coli* and *S. aureus* adhesion on coated samples.

Further attempts regarding polymer-based coatings deposited onto fixed orthodontic metallic components include: **(i) 2-methacryloyloxyethyl phosphorylcholine**: an hydrophilic and antibacterial phospholipid polymer with low friction property, which reduced the CoF of coated SS wires against brackets, and inhibited the in vitro adhesion of *S. mutans* and *P. aeruginosa* [510]; **(ii) butyl-3-methylimidazolium chloride**, which reduced the in vitro corrosion of NiTi archwires in fluoride- and/or bovine albumin-containing artificial saliva; **(iii) 1H,1H,2H,2H-perfluorodecyltrimethoxysilane** with bovine serum albumin, showing a non-bactericidal but highly antibacterial effect by preventing adhesion through a protein-mediated mechanism [513]; (**iv) polydopamine and carbon dots:** a fluorescent and antibacterial coating, with in vitro cytocompatibility, deposited on SS brackets [515]; **(v) polyoxazoline with tryptophan** coating, which decreased metal release and showed good in vitro biocompatibility to primary human dermal fibroblasts [512]; and **(vi) silicone** coatings on β-Ti and SS archwires [506].

5.2.2. Polymeric-Based Composite Coatings

Parylene polymer matrix reinforced with Ag and Pt is a commercially available coating on SS archwires [334,484,492]. The surface of the coated wires is typically rougher and softer, even though the bulk maintained its mechanical properties when compared with the uncoated ones [334].

Epoxy matrix reinforced with nanoscale TiO_2 particles (~200 nm) was studied by Xu et al. [517] on NiTi sheets and round wires. The researchers concluded that the tooth-like-colored coatings were thinner (~22 μm) and presented better surface quality (smooth, defect-free, and uniform) than commercially available aesthetic epoxy coatings (~50 μm). Stress–strain curves of both coated and uncoated archwires, from loading-unloading tensile tests at 8% pre-strain, almost overlapped, indicating that superelasticity and shape-memory properties remained unchanged. After a 30-day immersion test in Fusayama-Meyer artificial saliva, both coating microhardness and adhesion slightly decreased (yet statistically non-significant), while Ni release was effectively blocked without corrosion signs. Moreover, no cytotoxicity in MG-63 cells was detected. Results are, therefore, optimistic, even though further variables must be added to the experiment to better simulate the oral environment; for instance, a more corrosive medium, bracket/wire contact, and microbiological attack [517]. Zhou et al. spray-coated β-Ti and SS archwires with commercial ceramic-reinforced epoxy coatings [488].

Recently, Gholami et al. [442] successfully reinforced polymeric coatings with ZnO-NPs: (i) ZnO-NPs-containing polyvinyl pyrrolidone (PVP) by electrospinning; and (ii) ZnO-NPs-containing PVA by a polymer composite coating method. However, such coatings displayed less antibacterial efficacy than simple ZnO-NPs coatings (non-composites) even though all surfaces were antibacterial: the reduction percentages regarding *S. mutans* viability with non-composite ZnO-NPs coatings ranged between ~93 and 99%, whereas ZnO-NPs-containing PVP and PVA composite films showed values of ~72 and 90%, respectively [442].

A more complex polymeric-based composite coating system, which consisted of a 3-layered structure covered with Ag nanoparticles onto a flat SS substrate, was developed by Lee et al. [518] and is worth mentioning. The studied architecture can be seen in Figure 22. While the two poly(3,4-ethylenedioxythiophene)-based inner layers enhanced adhesion to the substrates, the outmost dopamine layer decreased bacterial adhesion for both *S. mutans* and *E. coli*. Adsorbed silver (Ag^+ and Ag-NPs) conferred antifouling and antibacterial properties without releasing toxic amounts of Ag species or compromising the in vitro cell viability of human gingival fibroblasts [518].

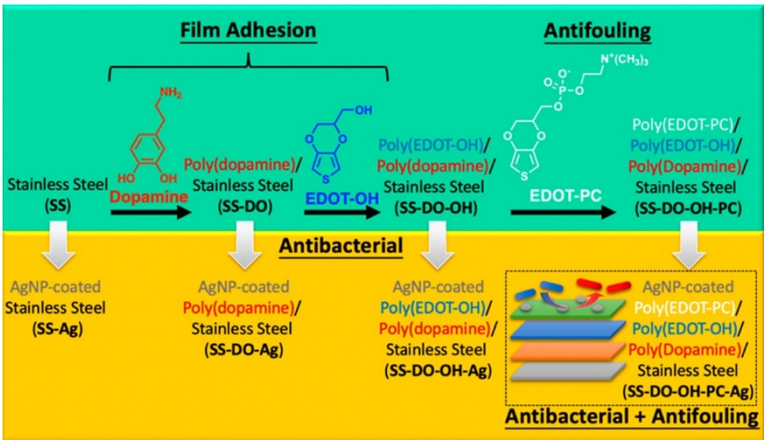

Figure 22. A complex polymeric-based composite deposited onto an SS flat substrate to confer the surface with antibacterial and antifouling properties (reprinted with permission from [518]. Copyright (2020) American Chemical Society).

5.3. Ceramic-Based Coatings

The authors chose to include within this section the studies covering calcium (Ca) and phosphorus (P)-based coatings, comprehending hydroxyapatite (HA) materials, and bioactive glass (Table 11). An overview of covalent C-based materials is also presented, including hydrogen-free amorphous carbon nitride (a-CN_x) and diamond-like carbon (DLC) coatings, as well as silicon-based coatings (SiN, SiC, and SiO_2-NPs).

5.3.1. Hydroxyapatite (HA)

Hydroxyapatite (HA), that is, pentacalcium hydroxyl apatite [$Ca_{10}(PO_4)_6(OH)_2$], is a mineral frequently used in many biomedical applications, namely bone-related conditions [519], due to its excellent biocompatibility and osteoconductive properties. Moreover, HA is now commercially available in oral care products [520], and some attempts were made to use it as a surface material for orthodontic components.

Jiang et al. [521] coated porous SMA–Nitinol alloy with HA by the SHS method, doing a 5-day immersion in Hank's solution after NH_3 and NaOH aqueous solution treatments. The resulting bone-like HA layer reduced, by one order of magnitude, the Ni release after a 50-day body fluid immersion test. Kocijan et al. [522] confirmed the barrier property of the HA coatings: The electrodeposited layer effectively enhanced corrosion resistance in Hank's solution of NiTi samples. Even a thin amorphous calcium phosphate layer (~600 nm thick) deposited by r.f. magnetron sputtering reduced nickel release from NiTi plates [523].

Unfortunately, only two studies were found using real orthodontic components. Setiyorini and Pintowantoro [414] synthesized biomimetic HA on NiTi wires by electrodeposition, revealing the highest cell viability percentage compared to other studied coatings. Dimasruhin et al. [524] deposited a SiO_2-containing HA coating via electrodeposition. The researchers successfully coated NiTi archwires but only performed a morphological characterization.

The evident lack of studies dealing with metallic orthodontic components (brackets, archwires) is probably related to the ceramic-brittle nature of HA.

5.3.2. Bioactive Glasses

Bioactive glasses have also been studied by Kawaguchi et al. by deposition on both SS disks [525] and archwires [526] through an electrophoretic process under 10 or 15 V (direct or alternating current: d.c. or a.c., respectively). Coatings (1–4 μm thick) onto SS disks showed promising aesthetics, non-cytotoxicity, and remineralization ability up to 3 months of acid etching [525]. Archwires coated at 10 V demonstrated significantly higher friction against SS brackets up to 10° of bracket/archwire angulation [526]. This can be due to the lower coating hardness when compared with the metallic surface (<0.5 vs. 6.11 GPa, respectively). On the other hand, coatings deposited at 15 V, with a hardness value of ~2 GPa, provided similar friction as unmodified wires against SS brackets. The main downside reported was the disruption/detachment behavior in the bracket/archwire interface during the friction tests [526].

5.3.3. Silicon-Based Coatings

SiN and SiC-based coatings were deposited on SS brackets and archwires by Rapiejko et al. [527] in an attempt to reduce both friction and wear during the sliding of those orthodontic components. Those authors designed an ex-situ fretting test to allow micro-sliding (simulating the occlusion movements) to superimpose on macro-sliding (which in turn simulates the global displacement of the teeth), both in dry and wet (artificial saliva) environments. The lowest friction and the highest wear were found for the uncoated SS bracket and archwire contact, whereas the opposite was true for the coated pairs. The researchers concluded that due to such a higher CoF (0.50 vs. 0.20 for coated and uncoated pairs, respectively, in artificial saliva), and despite the lower wear, the coatings are unsuitable for medical applications [527]. Silveira et al. [449] also reported a decrease in surface roughness and friction forces in both dry and artificial saliva environments when SS orthodontic wires were coated with an SiO_2-NPs-based film, when compared with uncoated ones, against brackets.

5.3.4. Carbon-Based Coatings

- C and C–H Systems:

Single C and binary C–H systems are very broad groups of materials, encompassing the non-hydrogenated and hydrogenated amorphous carbon—a-C and a-C:H, respectively. Usually, these materials are ambiguously recognized as diamond-like carbon (DLC) in the literature [528,529], despite their variable C=C sp^2 (similar to graphite) and C-C sp^3 (similar to diamond) hybridization bondsand H content. DLC coatings are widely used in the medical field [530–533]. The major advantages of DLC coatings are their outstanding overall biocompatibility, corrosion resistance, achievable high hardness, and low CoF. For further knowledge on the DLC materials, it is recommended to read the well-known review by J. Robertson [534]. To the authors' best knowledge, there is yet no commercial application in fixed orthodontics, and the first attempts to use DLC coatings on real orthodontic components date back to 2005 (Table 11). Studies focused on coating brackets and archwires with DLC are hereafter described.

Kobayashi et al. [535,536] were able to reduce the in vitro corrosion of NiTi archwires. The researchers successfully deposited a DLC coating (presumably a-C:H) with an SiC interlayer by ion beam plating with benzene as the reactive gas. The coating was stable for a 24 h continuous mechanical brushing and significantly decreased the Ni release in physiological saline solution up to 14 days (short-term, 80 °C) and 6 months (long-term, 37 °C) when compared with uncoated NiTi archwires. In all, the DLC coating showed excellent adhesion and mechanical properties to mechanical brushing and prevented corrosion [535]. In two quite similar reports [537,538], the ~1 μm thick a-C:H coatings—deposited by an arc discharge ion plating method with benzene—reduced Ni release from NiTi archwires by 80% in a 5-day static immersion test (physiological saline solution, 85 °C).

Simulating basic intraoral conditions is essential, but the corrosion susceptibility of orthodontic alloys increases in fluoride-acidic environments [29,42,95,104,276,277]—and

daily-used mouthwashes and toothpastes include fluoride prophylactic agents in their compositions. Accordingly, the corrosion protection ability of a 100 nm thick DLC (likely a-C) film deposited by mirror-confinement-type electron cyclotron resonance (MCECR) plasma sputtering on NiTi archwires was studied against a NaF-based anticavity dental rinse (pH 4) [539]. All samples were statically immersed in an artificial saliva for 12 weeks at 37 °C, and additionally dipped for 5 min in a mouthwash (0.044% sodium fluoride, in acidulated phosphate solution) three times a day. While the surface roughness of uncoated samples increased by ~50% due to F-induced dimples and cracks, the coatings protected the substrate against F-induced corrosion, with only a slight increase in surface roughness (~8%) and no significant morphological alterations [539].

To reduce resistance to sliding, some researchers deposited a-C:H coatings onto orthodontic brackets [423,539–542] and archwires [535,537–539,542–547], or even both components [548]. Deposition techniques included Plasma-based ion implantation/deposition (PBIID) [540,542,544,547], MCECR plasma sputtering [539,548], arch-discharge ion plating [535,537,538,545], and PECVD [541,543,546]. To promote and evaluate bracket/wire sliding properties, such as frictional forces and friction coefficient, custom-made devices are usually selected.

Tantiwinyupong et al. [547] found significantly different static frictional forces between conventional and DLC-coated NiTi archwires against brackets (dry, room temperature 25 °C), representing a decrease of ~33%. The PBIID-deposited a-C:H coating also improved surface hardness from 1.06 to 11.44 GPa. Similar results were reported by Muguruma et al. [542] for NiTi and SS wires coated by PBIID against uncoated conventional or self-ligating brackets, up to a 10° angulation at room temperature and in a dry environment. The 500 nm thick a-C:H layer increased surface hardness from 11.6 to 17.6, and from 4.7 to 9.1 in the case of SS and NiTi, respectively. Thicker a-C:H coatings (~5–7 μm) produced by the same technique on SS brackets can also reduce both friction forces against SS brackets under dry and wet conditions [540].

Zhang et al. [546] produced a-C:H coatings (~1 μm) on SS wires by PECVD, obtaining a DLC surface smoother and 1.46 times harder than the uncoated wire. However, no statistically significant differences between the static friction coefficient or maximum static friction force were observed under dry conditions—only the kinetic friction coefficient decreased by ~40%. Kang and colleagues [548] also reported a decrease in the kinetic friction between SS wires and brackets. They tested MCECR plasma sputtering-produced, 200 nm thick a-C:H coatings, obtaining a reduction in the kinetic friction coefficient by ~80 and 70% under ambient air and artificial saliva, respectively, when at least the wire was coated. The lowest value of CoF = 0.11 was reported for both components under ambient air. Although kinetic friction seems irrelevant in orthodontics [215], the authors argued that their test involved oscillations with small displacements (±150 μm, 0.5 Hz), which recreated the discontinuous clinical motion (5 mm of distance, 10 mm/min) [548], in opposition to others [423,540,542].

Danisman et al. [549] deposited an 83 nm thick DLC top-coating above a Ti/TiN coating (36/687 nm) film on SS brackets by closed-field magnetron sputtering and measured the static friction force against SS archwires in air (5 mm of distance, 10 mm/min). The researchers reported a 28–39% decrease in the friction forces when coatings were used in comparison with uncoated brackets [549].

S. Huang et al. [539] coated NiTi archwires with a thin 100 nm a-C:H layer by PECVD. Surface roughness did not significantly change, but bracket/wire friction coefficients diminished up to 79.7% and 70.0% in ambient air and artificial saliva, respectively, when compared with uncoated tribo-couples. In a static immersion test simulating fluoride mouth rinse, surface roughness variations—caused by fluoride-induced corrosion—decreased by 91.3%, demonstrating the anti-corrosion behavior of amorphous carbon films [539].

The general tribological improvement by using a-C:H coatings may be related to the expected increase in surface hardness, which leads to a lower wear rate and, therefore, lower frictional forces [542]. Higher surface hardness is advantageous for orthodontics,

as it may minimize binding or notching effects [542,547]. Muguruma et al. [544] pointed out the role of H on the a-C:H surface, suggesting that DLC coatings with high H content (>30 at.%) are potential candidates for this application.

Surface wettability may also play another important role in reducing static friction [541,543]. To further explore this idea, Akaike et al. [541] deposited unmodified and F- and Si-modified a-C:H coatings on SS bracket slots by PECVD. Under wet (PBS-sprayed) conditions, all coatings decreased static friction when compared with uncoated bracket slots, up to a bracket/wire angulation of 10°. However, a-C:H:F coatings showed the lowest static friction. This may be due to the higher hydrophobicity of such coating, reflected by the highest PBS and distilled water contact angles (~80 and 90°, respectively) when compared to the SS surface (~60°) and even to the a-C:H film (~70 and 75°). The a-C:H:Si films were more hydrophilic than the other two DLC-based coatings (contact angles of ~35°), and therefore the decrease in static friction was lower [541].

Mechanical brushing with fluorine-containing toothpaste is an indispensable daily hygiene procedure, especially for patients undergoing fixed orthodontic treatment [29,91,108]. Therefore, Oghoe, Kobayashi et al. studied the stability of 1 µm thick a-C:H coatings deposited on orthodontic NiTi archwires by arch-discharge ion plating with [535] and without [537,545] a SiC interlayer. The researchers simulated a 6-month daily procedure by mechanically brushing the coated wires with equipment carrying a toothbrush for 250 min, 350 min, and 24 h at a constant load of 35 g. The coatings protected the substrate that corroded when uncoated; no peeling, cracks, or even microstructural changes were detected, indicating good coatings' adherence and stability against brushing hygiene procedures [535,537,545].

Designing coatings with antibacterial properties—for instance, by reducing bacterial adhesion—can minimize the formation of biofilm layers [550]. Currently, the eventual antibacterial properties of DLC coatings are non-consensual, even though the introduction of antibacterial agents is possible [551]. Furthermore, and to the authors' best knowledge, little investigation has been conducted using amorphous carbon coatings specifically for orthodontics [550,552], and no study was found assessing the antibacterial properties of DLC-coated fixed orthodontic components. Further research is required to assess and enhance the antibacterial properties for orthodontic applications.

- C–N System:

Carbon nitride (CN_x) coatings are well-recognized biocompatible and chemically stable surface materials with attractive mechanical and tribological properties for biomedical applications. Following previous research [553], Wei et al. [554] coated SS orthodontic archwires with CN_x by ion beam assisted deposition (IBAD). Published results show lower and more stable frictional forces and CoF under both ambient air and artificial saliva, especially at higher bracket/wire angulations (up to 15°). Compared with bare or TiN-coated SS304L disks, those IBAD-produced CN_x thin films (<500 nm) presented the best surface properties, that is, a lower and more stable coefficient of friction (CoF < 0.2), biocompatibility with human fibroblasts and effective antibacterial properties against *S. mutans* [459].

Table 11. Overview of ceramic-based coatings deposited on different orthodontic appliances and substrates. CVD: chemical vapor deposition; PECVD: plasma-enhanced CVD; PBIID: plasma-based ion implantation and deposition; IBAD: ion beam-assisted deposition; MCECR: mirror-confinement-type electron cyclotron resonance.

Coatings	Substrate Materials	Deposition Methods	Literature References
Hydroxyapatite and Bioactive Glass Coatings			
Bioactive glass	SS archwires	Electrophoretic deposition	[526] Kawaguchi et al. (2020)
SiO_2-reinforced HA	NiTi archwires	Electrodeposition	[524] Dimasruhin et al. (2014)
HA	NiTi archwires	Electrodeposition	[414] Satiyorini and Pintowantoro (2013)

Table 11. *Cont.*

Coatings	Substrate Materials	Deposition Methods	Literature References
Silicon-based Coatings			
SiN	SS brackets and archwires	PECVD	[527] Rapiejko et al. (2009)
SiC	SS brackets	PECVD	[527] Rapiejko et al. (2009)
SiO_2-NPs	SS archwires	Commercial ceramic paint	[449] Silveira et al. (2022)
Carbon-based Coatings			
DLC (a-C:H)	NiTi archwires	Ion beam plating	[535] Kobayashi et al. (2005)
DLC (a-C:H)	NiTi archwires	CVD	[536] Kobayashi et al. (2005)
DLC (a-C:H)	NiTi archwires	Arch discharge ion plating	[538] Ohgoe et al. (2006)
DLC (a-C:H)	NiTi archwires	Arch discharge ion plating	[537] Ohgoe et al. (2007)
DLC (a-C:H)	NiTi archwires	Arch discharge ion plating	[545] Kobayashi et al. (2007)
DLC (a-C:H)	SS brackets	PECVD	[423] Huang et a. (2010)
DLC (a-C:H)	NiTi and SS archwires	PBIID	[542] Muguruma et al. (2011)
DLC (a-C:H)	SS brackets	PBIID	[540] Muguruma et al. (2013)
DLC (a-C:H)	NiTi archwires	PECVD	[539] Huag et al. (2013)
DLC (a-C)	SS archwires	MCECR plasma sputtering	[548] Kang et al. (2015)
DLC (a-C:H)	SS backet slots	PECVD	[543] Akaike et al. (2015)
DLC (a-C:H)	SS bracket slots	PECVD	[541] Akaike et al. (2016)
DLC-F (a-C:H:F)	SS bracket slots	PECVD	[541] Akaike et al. (2016)
DLC-Si (a-C:H:Si)	SS bracket slots	PECVD	[541] Akaike et al. (2016)
DLC (a-C:H)	SS archwires	PECVD	[546] Zhang et al. (2016)
DLC (a-C:H)	SS archwires	PBIID	[544] Muguruma et al. (2018)
DLC (a-C:H)	NiTi archwires	PBIID	[547] Tantiwinyupong et al. (2019)
DLC (a-C)	SS brackets	Magnetron sputtering	[549] Danisman et al. (2021)
CNx	SS archwires	IBAD	[554] Wei et al. (2011)
Carbon-based Nanocomposite Coatings			
Graphene sheets embedded in carbon	SS archwires	MCECR plasma sputtering	[555] Pan et al. (2022)
Graphene sheets embedded in carbon	SS archwires	MCECR plasma sputtering	[556] Wang et al. (2022)

- Carbon-based Nanocomposite Coatings:

Graphene sheets embedded in carbon (GSEC) matrixes are a recently deposited material on SS archwires by Pan and coauthors [555,556] via MCECR plasma sputtering. The aim was to decrease the CoF against SS brackets in wet conditions (Fusayama–Meyer artificial saliva). The researchers reported the lowest CoF and wear rate of 0.05 and 0.11×10^{-6} mm^3/Nm when sliding against three-row micro-groove-textured SS brackets (10,000 times, 150 μm displacement stroke, 0.5 Hz, and 0.5–2.0 N). They proposed that the excellent friction and wear performances are due to the formation of a tribofilm on the contact interface formed by an adsorbed saliva layer and graphene sheets, coupled with the removal of debris with artificial saliva through the micro-grove (Figure 23) [555,556].

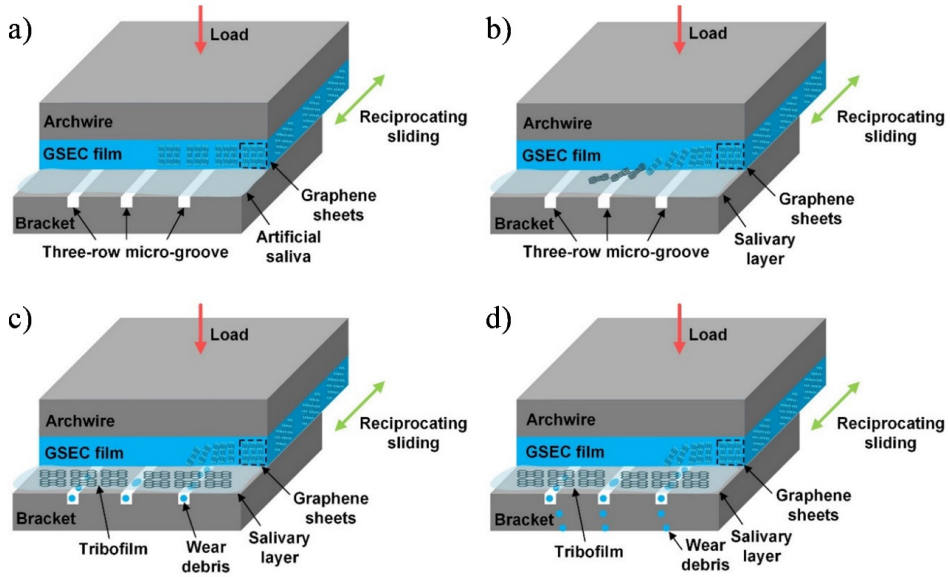

Figure 23. Low friction mechanism of graphene sheets embedded carbon (GSEC) coating on an SS archwire against an SS bracket: (**a**) initial state of the contact combination; (**b**) stable low friction with the formation of graphete-rich tribofilm and salivary adsorbed layer; (**c**) accumulation of wear debris detached from the GSEC film with micro-groove; and (**d**) flow out of wear debris with artificial saliva from the micro-groove (reproduced from [556]).

6. Conclusions

Contemporary orthodontics heavily relies on using fixed orthodontic appliances to treat dental malocclusions. Those devices are commonly manufactured using metallic bioalloys due to their suitable mechanical properties that induce the necessary tooth movement through the treatment time—usually around 2 years. Stainless steels and nickel–titanium stand out due to their widespread use, followed by CoCr- and other Ti-based alloys.

The main downside of using metallic biomaterials in vivo is the inevitable corrosion process in biological environments. In fact, the human mouth is an extreme corrosion-promoting scenario, encompassing frequent and complex variations in chemical composition, temperature, and pH, promoted by many factors such as diet, oral microorganisms, and the use of F^--containing hygiene products. Consequently, several types of corrosion could occur, including pitting, crevice, fretting, and so-called microbiologically induced corrosion.

The main consequences of intraoral corrosion are presented in this overview. The release of metallics is the most frequently studied feature; 67 studies measured the metal content in various matrices. While concentrations of released metal ions are below toxic levels, reported allergic reactions to metals such as Ni during orthodontic treatments with fixed appliances raised flags. This topic is not new, but further clarification is still required. Moreover, corrosion can increase the bracket/archwire pair friction, which may make it difficult to determine the correct treatment progression. The use of fluoride-containing products—unquestionably important to maintain oral health—was also focused, as they can potentially aggravate metallic corrosion susceptibility.

The literature shows several paths that researchers have been following to improve the overall corrosion resistance and, in consequence, the biocompatibility of orthodontic alloys. Those include the replacement of metallic components with non-metallic or composite substitutes; the modification of the alloys' chemical composition; the development of different manufacturing processes; and the application of surface modification techniques by depositing protective coatings. The versatile use of thin films and coatings stands out in this biomedical field and is particularly focused on this review, as it can maintain the crucial properties of the alloys' bulk. Many different deposition technologies and materials

have been used in multiple in vivo and in vitro efforts to protect against oral aging, from monolithic to composite architectures and micro- to nano-scale materials, to meet the best and safest oral practice demands. Unfortunately, the challenging oral environment still undermines the development of a fully effective coating, whereas the high variability of research methodologies challenges the direct comparison between the studies.

Data suggest that even existing commercially available alternatives have important drawbacks and are fallible. Further multidisciplinary research should, therefore, persist, namely by applying new coating materials from the surface engineering field to fixed orthodontics and conducting broader studies to mimic the intraoral cavity complexity.

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Abbreviations

AAS	Atomic absorption spectrometry
a-CNx	Amorphous carbon nitride
AISI	American Iron and Steel Institute
CAW	Composite archwires
CVD	Chemical vapor deposition
CoF	Coefficient of friction
d.h.m.	Dry hair mass
DL	Detection limit
DLC	Diamond-like carbon
EDS	Energy-dispersive spectroscopy
FCC	Face centered cubic
GSEC	Graphene sheets embedded carbon
HA	Hydroxyapatite
HGF	Human gingival fibroblasts
IARC	International Agency for Research on Cancer
IBAD	Ion beam-assisted deposition
ICP-AES/ICP-OES	Inductively coupled plasma-atomic/optical emission spectrometry
ICP-MS	Inductively coupled plasma mass spectroscopy
IF-NPs	Inorganic fullerene-like nanoparticles
ISO	International organization for standardization
MAO	Micro-arc oxidation
MCECR	Mirror-confinement-type electron cyclotron resonance
MIC	Microbiologically induced corrosion
NPs	Nanoparticles
PC	Polycarbonate
PECVD	Plasma-enhanced chemical vapor deposition
PVD	Physical vapor deposition
PE	Polyethylene
PEEK	Poly-ether-ether-ketone
PEN	Polyethylene naphthalate
PET	Polyethylene terephthalate
PEO	Plasma electrolytic oxidation

PH	Precipitation hardening
PIIID	Plasma immersion ion implantation and deposition
POM	Polyoxymethylene
PP	Polyphenylene
PTFE	Polytetrafluoroethylene
PU	Polyurethane
PVA	Polyvinyl alcohol
PVP	Polyvinylpyrrolidone
SCC	Stress corrosion cracking
SEM	Scanning electron microscopy
SMA	Shape memory alloy
SRB	Sulphur reducing bacteria
SS	Stainless steel
XRF	X-ray fluorescence

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