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Materials used for suprastructures in oral rehabilitations with the help of implants

DOCTORAL THESIS ABSTRACT

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1. Summary of the main ideas presented in the doctoral thesis

1.1. Introduction

The trend of evolution in the field of dental medical industry is rapid. The paradigm shift towards biological techniques and materials with durable mechanical and functional properties, adapted and integrated to develop natural human connections, and even those that are meant to be as originating from or part of the human body (human bone for bone augmentation and stem cells for dental bud growth) is strongly felt as a liberation.

The years when toxic metals were used are coming to an end, a stage that was otherwise extremely necessary for the evolution of dentistry. Well-known amalgam restorations with high concentrations of mercury (51%) are still present in many patients. Metal and mercury intoxications have been the subject of major research in German schools and have revealed vital information regarding the degree of poisoning and the relationship between metal poisoning and the human body [1]. Initially, the question was whether various intoxications indeed came from amalgam fillings. Toxicologists discovered that ultimately, mercury intoxication does not have significant incidence in the blood or urine but is synthesized in the brain, kidneys, and liver. Many forms of cancer are correlated with the release of mercury particles [2] [3].

I appreciate that research related to amalgam fillings was an essential starting point for opening horizons to the study of metals used in prosthodontic suprastructures. There is a clear international interest in this field, and for these reasons, international dental practice relies predominantly on the use of biomaterials.

Thus, this field is still new for many specialists who are just discovering the effects that metals, compounds, and cements have on human health [4].

The anatomical changes caused by edentulism, whether single-tooth, partial, or complete, require different approaches to restoring lost functions. The variations in prosthodontic rehabilitation using implants are determined based on the existing clinical situation and the correlation of information obtained through paraclinical investigations. Implant-prosthetic treatment aims to restore edentulous structures from a biological, biomechanical, occluso-articular, neuro-muscular, and aesthetic standpoint [5].

The rehabilitation of edentulism, the determination of implant types, and the measurement of receptive bone areas are done following a diagnosis and treatment plan.

After obtaining medical history, clinical and paraclinical analyses, it can be determined whether the patient is eligible for implant surgery [6] [7] [8] [9].

The implant-prosthetic structure can be used for both single-tooth and extensive, sub-total/total edentulous cases, through the creation of fixed or removable prosthodontic works [10].

The materials used for the fabrication of prosthetic structures on implants interact with the human body and are inserted into a living system to communicate with it and integrate, with the purpose of recreating the lost physiological conditions.

Each case requires an adapted solution, with materials having different properties, structures, alloys, without causing wear over time or generating negative effects on the human body.

For the fabrication of prosthodontic works with implant integration, various categories of materials are used, such as ceramic materials, polymers, noble and non-noble metal alloys, binary Ti-Zr systems, and innovative biomaterials like BioHpp, graphene, etc. [11] [12].

1.2. Research methodology

For the elaboration of the general part of the doctoral thesis, I conducted a synthesis of the specialized literature at the national and international levels, which provided support for the preparation of the work and helped in better supporting the presented arguments. As a result, the doctoral thesis has an interdisciplinary character as it involved interaction with the field of statistics, materials engineering, dental techniques, toxicology, allergology, immunology, and biophysics.

The personal part begins with a comparative study between two metallic alloys, Cr-Ni (Ni-Cr-Mo), and Cr-Co (Co-Cr-Mo-W), frequently used in prosthodontic suprastructures, where I aimed to evaluate the compositional changes that occur after repeated castings using the same crucible and the reuse of the remaining alloys after casting.

Starting from the study of a Ti-18Zr binary alloy available on the commercial market, I deepened my research through laboratory analyses on metal alloys, with the purpose of determining the reliability of using a higher concentration of zirconium - 20Zr - in the alloy with titanium for implant-prosthetic rehabilitation. Therefore, I conducted a comprehensive characterization of the potential biocompatible alloy from the Ti-20Zr binary system.

Finally, I addressed the method of applying a questionnaire-based survey on a sample of 55 dentists. Their responses were statistically analyzed using the Jamovi software, applying the chi-square test of correspondence, and the answers were also interpreted quantitatively and qualitatively.

1.3. Research hypotheses and objectives

Following the analysis of the specialized literature, I concluded that among the most commonly used methods of prosthetic suprastructures on implants are those based on metal alloys. Scientific studies frequently address the effects of materials used in suprastructures on the human body. The release of metal ions resulting from the interaction of metal alloys in the presence of salivary electrolyte generates toxic effects on living tissues, including allergic and carcinogenic reactions, among others. Moreover, the metals present in the composition of the metal alloy have unwanted/harmful effects on the human body. Considering the phenomena that occur in this interaction between metal alloys in the oral cavity, I aimed to create a thesis that addresses laboratory studies to scientifically argue the causes of the previously mentioned effects. Therefore, I structured the research hypotheses based on the following statements:

A. By using classical casting methods for some of the most commonly used metal alloys in the dental laboratory, namely Cr-Ni and Cr-Co, compositional changes occur as a result of crucible reuse, as well as in the case of using remelted alloys.

B. By adding a high percentage of 20% zirconium oxide to the Ti-Zr binary alloy, the structural, physical, mechanical, and chemical properties are improved compared to the Ti-V-Al alloy and the products available on the commercial market.

C. The approach to the methods and techniques for creating materials used in prosthetic suprastructures on implants is not a widely discussed subject between dental technicians and dentists; although the range of materials for prosthetic suprastructures is increasing, and innovative materials have emerged, dental practices still widely use classical methods of metal structure with various coatings.

1.3.1. The research objectives are directly related to the purpose of the doctoral thesis and each hypothesis:

Objectives for hypothesis A:

1. Laboratory testing of the physical and chemical properties of Cr-Ni and Cr-Co material alloys that have been subjected to a recycling process through repeated castings and repeated use of crucibles.

Objectives for hypothesis B:

1. Laboratory analysis of a Ti-Zr binary alloy with a higher composition of zirconium, specifically 20% Zr.
2. Establishing the processing method for the alloy.
3. Determining the physical and mechanical properties of the resulting alloy and evaluating it based on predetermined criteria.
4. Evaluating the reliability of using the resulting material in oral restorations.

Objectives for hypothesis C:

1. Developing questions aimed at identifying the variables contained in the hypothesis.
2. Distributing a questionnaire to a predetermined number of dentists.
3. Qualitative and quantitative interpretation of the responses and statistical modeling of the data.

2. Synthesis of the doctoral thesis chapters

The doctoral thesis is structured into two parts as follows:

The general part comprises data from the specialized literature on the addressed topic and is structured into two chapters.

Chapter 1 – Aspects of Implant-Prosthetic Rehabilitation - includes theoretical details in subchapters regarding modifications in anatomical areas in partial edentulism, modifications in anatomical areas in total edentulism, implant-prosthetic treatment, general aspects of the surgical stage for dental implant insertion, prosthetic abutments, general aspects of the prosthetic stage for the fabrication of implant-supported restorations, and variants of prosthetic suprastructures on implants.

Chapter 2 - Materials used in Dental Implant Suprastructures - addresses aspects related to the categories of materials most commonly used in the dental laboratory for implant suprastructures and innovative biomaterials.

The Personal contribution part contains the contribution made through original studies and is structured into three chapters, namely chapters 3, 4, and 5, where the conducted studies and the conclusions derived from them are presented. These chapters will be further detailed. The conclusions and original contributions are presented in the contents of chapter 6.

Chapter 3 - Studies on the influence of casting process of alloys used in implant-prosthetic restorations on chemical composition and microstructural characteristics

3.1. Material and method

In this study, we investigated the compositional changes that occur as a result of using new and old crucibles and the situation of using remelted alloys.

We analyzed two alloys commonly used in current practice for implant-prosthetic restorations: SOLIBOND N (Ni-Cr) and SOLIBOND C plus (Co-Cr).

The tests targeted metal pellets as they are distributed on the market, alloys after the first casting, alloys after casting in a mixture of 50% new alloy/ 50% remelted alloy, as well as alloys cast by remelting 100% of the remnants from previous castings. For better comparison, new ceramic crucibles were used, as well as reused ceramic crucibles, following the scheme presented in Table III. 1.

Table III. 1

The diagram of the pieces subjected to analysis

Ni-Cr – metal pellets		*Co-Cr – metal pellets*	
Ni-Cr – New crucible First casting	*Ni-Cr – Reused crucible First casting*	*Co-Cr – New crucible First casting*	*Co-Cr – Reused crucible First casting*
Ni-Cr – New crucible Mixed casting ½ Cone, ½ new	*Ni-Cr – Reused crucible Mixed casting ½ Cone casting, ½ new*	*Co-Cr – new crucible Mixed casting ½ Cone, ½ new*	*Co-Cr – reused crucible mixed casting ½ Cone, ½ new*
Ni-Cr – New crucible Casting	*Ni-Cr – Reused crucible Mixed Casting*	*Co-Cr – new crucible Casting*	*Co-Cr – reused crucible Mixed casting*

The preparation of the test pieces (melting and casting of alloy samples) was carried out within the Discipline of Prosthetic Technology and Dental Materials, "Carol Davila" University of Medicine and Pharmacy, Bucharest.

The material testing was conducted at the Center for Research and Expertise in Special Materials, the Laboratory of Spectrochemical Emission and X-ray Fluorescence Testing, within the Faculty of Materials Science and Engineering (Dept. SMMMF), Polytechnic University of Bucharest.

3.2. Results

3.2.1 Results obtained on the Ni-Cr alloy (Ni-Cr-Mo)

Spectrometric analysis

Elemental concentrations were measured using the SpectromaxX spectrometer with the Ni-base analytical program. Alloy pellets, as provided by the manufacturer, were analyzed to have a reference element to which we compared all measurements made on the samples established for the study (Table III. 2).

Tabel III. 2 Mass concentrations for the reference sample Ni-Cr (metal pellets) (%)

Ni	*Cr*	*Mo*	*Si*	*Fe*	*Ta*	*Co*	*Cu*	*Nb*
61.983	*26.45*	*9.75*	*1.18*	*0.530*	*0.110*	*0.0*	*0.0*	*0.0*

The values obtained through the analysis of the alloy pellets were compared with those from the product data sheet provided by the manufacturer to determine if there are significant differences in the component elements. Some differences can be observed, but they are not significant. The only differing element from the manufacturer's data sheet is Tantalum, which is present in the analyzed metal pellets.

Element analysis for the Ni-Cr pieces obtained using new crucibles shows variations in Si and Cr compared to the reference values for all mixing variants (casting from new alloy, casting from a mixture, and casting from the cone).

Depending on the number of standard deviations between the first casting and casting from a mixture, we find uncertainties for Molybdenum, which is slightly in excess for both variants, and for Chromium, the quantity of which decreases. Silicon shows a slight increase for casting from a mixture, but the value is more significant in the case of casting from the new alloy (4σ).

By comparing the results between the first casting and casting from the cone, we see the same minor variations for molybdenum and chromium. However, Silicon, in both cases, is in excess, with 4σ for the first casting and 4.25σ for casting from the cone.

Tabel III. 3

Mass concentrations for the Ni-Cr samples made with a reused casting crucible

Component elements	*First casting*		*Mixed casting*		*Cone casting*	
	Value	*Standard deviation*	*Value*	*Standard deviation*	*Value*	*Standard deviation*
Ni	*60.46*	*1.6*	*62.2*	*1.52*	*61.925*	*1.27*
Si	*1.320*	*0.03*	*1.810*	*0.05*	*1.330*	*0.03*
Cr	*24.500*	*0.80*	*22.620*	*0.80*	*23.690*	*0.70*
Mo	*10.100*	*0.60*	*10.120*	*0.50*	*11.060*	*0.40*
Fe	*0.480*	*0.05*	*1.000*	*0.04*	*1.260*	*0.03*
Ta	*0.120*	*0.02*	*0.110*	*0.03*	*0.115*	*0.02*
Co	*2.270*	*0.06*	*0.220*	*0.04*	*0.500*	*0.05*
Cu	*0.750*	*0.04*	*1.920*	*0.06*	*0.000*	*0.00*
Nb	*0.000*	*0.00*	*0.000*	*0.00*	*0.120*	*0.04*

Comparing the results between the first casting and casting from a mixture, we find uncertain variations regarding Cr, which is slightly deficient (-2.44σ), and Fe, which is slightly in excess (1σ) for the piece from the first casting. For the first casting, significant values are observed for Si (4.67σ), but the most significant values that raise questions are related to elements present in excess, Co (37.83σ), and Cu (18.75σ), which should not be part of the alloy composition. The casting from a mixture shows significant values for Cr, which is even lower compared to the first casting (-4.79σ), and significant increases for Si (12.60σ) and Fe (11.75σ). Significant values are also found for Co, which is present despite not being expected (5.50σ), and the most significant increase is observed for Cu (32σ).

Analyzing the first casting (from new alloy) and casting from the cone (100% remelted alloy), we observe the same loss of Cr at a significant value (-3.94σ) and an increase in the quantity of Mo (3.28σ). The increase in the amount of Si (5σ) and Fe (24.33σ) is maintained. Additionally, compared to the reference values, Co (10σ) is present, and in smaller quantities, Niobium (3σ).

When a new crucible is used, variations from the reference values are small, with the most significant being for Cr and Si (approximately 4σ).

The large variations that occur when a reused crucible is used can be explained by the alloy film that remains on the internal surface of the crucible from one casting to another, which cannot be entirely cleaned without damaging the crucible. Significant quantities of Fe, Co, and Cu remain. What seems to be more important is the preferential combination of the Ni-Cr alloy with Cu residues. During casting from a mixture, the largest quantity is obtained, which is a combination of the 2nd and 3rd casting stages, and in the last two stages, Cu is absent because it is not present in the casting from the cone (100% remelted alloy), which is the final casting stage.

Additionally, quantities of Co, not specified in the manufacturer's data sheet and not found in the analysis of samples using new crucibles, are present. The degree of combination with the Ni-Cr alloy is higher in the first casting, decreases during casting from the mixture, and slightly increases during casting from 100% remelted alloy. This indicates persistence in the remnant alloy film on the crucible walls and a low affinity of the Ni-Cr alloy to absorb Co residues. Fe is also noteworthy. In the first casting using a reused crucible, it undergoes a slight decrease, but with repeated castings, it increases up to 24.33σ. It is possible that during repeated castings, at short intervals, Fe is entrained from the remnant film, with the final casting stage combining residues from multiple recastings.

Qualitative analyses of optical microscopy and metallographic etching were also performed in the study, revealing the presence of non-uniformly distributed intermetallic compounds. When using an old crucible, it was expected to have more inclusions of different sizes due to the variety of chemical compositions resulting from the reuse of crucibles. At the dendritic structure level, it appears as a continuous chain when using a new crucible, while in the case of using an old crucible, this chain seems to be interrupted in some areas.

3.2.2 Results obtained for the Co-Cr alloy (Co-Cr-Mo-W)

Spectrometric analysis

In this case as well, elemental concentrations were measured using the SpectromaxX spectrometer, with the Co-base analytical program. The analysis of the metal pellets for the

Co-Cr alloy showed the following values for the base and complementary elements:

Tabel III. 4

Mass concentrations for the reference sample of Co-Cr (metal pellets) (mass %)

Co	*Cr*	*Si*	*Mn*	*Mo*	*Fe*	*W*	*Nb*
63.25	*21.97*	*0.67*	*0.18*	*3.68*	*0.23*	*9.8*	*0.22*

Comparing the data sheet provided by the manufacturer, we observe the presence of Mn and Iron in proportions lower than 1% in the values of the reference sample, compared to the standard formula.

Tabel III. 5

Mass concentrations for the Co-Cr samples made with a new casting crucible.

Component elements	*First casting*		*Mixed casting*		*Cone casting*	
	Value	Standard deviation	*Value*	Standard deviation	*Value*	Standard deviation
Co	*62.98*	*1.74*	*62.64*	*1.51*	*62.82*	*1.52*
Si	*0.72*	*0.03*	*0.71*	*0.03*	*0.72*	*0.03*
Mn	*0.16*	*0.02*	*0.17*	*0.01*	*0.16*	*0.02*
Cr	*22.40*	*0.90*	*22.18*	*0.90*	*22.34*	*0.90*
Mo	*3.62*	*0.40*	*3.74*	*0.30*	*3.70*	*0.30*
Fe	*0.26*	*0.06*	*0.25*	*0.05*	*0.16*	*0.04*
W	*9.65*	*0.30*	*10.09*	*0.20*	*9.89*	*0.20*
Nb	*0.21*	*0.03*	*0.22*	*0.02*	*0.21*	*0.03*

Analyzing the overall values obtained for the samples made using a new casting crucible compared to the reference values, we observe variations in Si, Mn, and Fe. Comparing the values for the samples obtained from the first casting and casting from a mixture using a new crucible with the reference values, we notice minor changes of up to 1.67σ. Thus, Si is slightly in excess, Mn is deficient (-1σ), and W shows an increase (1.45σ) for the casting from the mixture.

In the case of the piece obtained using remelted alloy, variations compared to the reference sample are less than 2σ. Si shows a slight increase (1.67σ), Mn is reduced (-1σ), and Fe is also decreased (-1.75σ). For the samples using a reused crucible, the following results were obtained from the spectrometric analysis:

Tabel III. 6

Mass concentrations for the Co-Cr samples made with a reused casting crucible.

Component Elements	First casting		Mixed casting		Cone casting	
	Value	Standard deviation	Value	Standard deviation	Value	Standard deviation
Co	62.8	1.64	62.91	1.62	62.903	1.53
Si	0.70	0.04	0.72	0.04	0.71	0.03
Mn	0.16	0.02	0.17	0.02	0.16	0.02
Cr	22.21	0.90	22.20	0.90	22.22	0.90
Mo	3.68	0.40	3.79	0.30	3.73	0.30
Fe	0.20	0.05	0.24	0.04	0.02	0.06
W	10.04	0.20	9.74	0.30	10.04	0.20
Nb	0.21	0.03	0.23	0.02	0.22	0.02

Comparing the reference values, variations are noticed for most of the analyzed elements, with the highest being for Fe in the case of the piece made from a remelted alloy. The analysis of the number of standard deviations showed, for the piece made from a new alloy, a decrease in Mn (-1σ) and an increase in W (1.2σ), both being statistically insignificant minor variations. For the piece made from an alloy mixture (50% new alloy and 50% remelted alloy), the only change was a slight increase in Si (1.25σ), also statistically insignificant. For the piece obtained from a remelted alloy, there are minor variations: an increase of 1.33σ for Si and 1.2σ for W, respectively, and a decrease of -1σ for Mn. The only element with a more significant variation is Fe, with a decrease of -3.5σ compared to the reference sample.

The analysis of the values obtained for the same type of mixture (100% new alloy, 50% new alloy/50% remelted alloy, and 100% remelted alloy) shows no statistically significant changes, with the only more significant variation being in Fe for the piece from the remelted alloy in a reused crucible, where a pronounced decrease (-3.5σ) was observed but at the limit of statistical sensitivity.

In the course of the studies, qualitative analyses of optical microscopy and metallographic etching revealed the presence of micropores and exogenous inclusions similar to those found in the Ni-Cr-Mo alloy system. Also, the presence of friable compounds is noticeable, and the trajectory of detached fragments during the last polishing stage can be traced, resulting in a visual effect of microscratches.

It can be asserted that the proportions of constituents and phases present in the alloy vary depending on the cooling rate and, consequently, the segregation degree of Cr, Mo, and W elements. With the increase in the number of recastings and the variation in the proportion of recycled alloy (remelted), other standalone phases may appear in the microstructure.

3.3. Discussions

The results obtained from the spectrometric tests and microstructural investigations on the Ni-based alloy clearly highlight the influence of the remelting process on the elemental composition modifications and microstructure variability that occur as a result of remelting.

In the case of Ni-based alloys (melted and remelted), a variation in chemical composition is observed, attributed to the tendency of different elements to segregate. Specifically, Cr tends to segregate at the surface of the melted alloys, while Co and W segregate between the surface and the lower part of the melted alloy, as observed in the paper "Surface tension of molten Ni-(Cr, Co, W) alloys and segregation of elements." [13]

The microstructural investigations have revealed, on one hand, the presence of inclusions resulting from insufficient degassing during their preparation (the number of inclusions increases when a reused crucible is used), and on the other hand, the formation of a dendritic structure (large dendrites with additional branches) with a continuous chain-like appearance (in the case of using a new crucible) and an interrupted chain-like appearance (dendrites are oriented in all directions, with similar large but less well-defined shapes) when a reused crucible is used. As a result, heterogeneous microstructures were obtained, which can influence the properties of the alloy when reused crucibles and recycled alloys are used.

These findings are consistent with those obtained by authors James J et al. and Vaillant-Corroy AS et al., who highlighted that a content of approximately 50% recycled alloy leads to modifications in the properties of the alloy, and the maximum number of remeltings should be limited to 4. Moreover, in the scientific research conducted by Al-Hiyasat AS and Darmani H, it was found that recycling/reusing metallic alloys has a negative effect on cytotoxicity, and the higher the content of recycled material, the higher the risk of element release and, consequently, an increase in toxicity. [14] [15] [16]

Furthermore, it is necessary to highlight the variations in elemental composition, specifically Fe, Cu, and Co, elements that were not part of the initial alloy but were present in the melting from the old crucible, with substantial variations between the second and third remeltings. The ternary alloy mainly forms a coalition with Cu.

In a scientific research regarding the effects of recycling base metal alloys on cytotoxicity, Al-Hiyasat AS and Darmani H. concluded that the presence of additional metals resulting from remelting with new and reused alloys leads to significantly higher toxicity due to the appearance of elements like Cu, among others. [16]

In the case of Co-based alloys, the results of the spectrometric tests show a small variation in the mass percentages of Fe, Mo, and W elements.

Chemical composition variation due to remelting can lead to the emergence of phases that modify the mechanical properties of the obtained alloy and may negatively impact the corrosion resistance of the alloy, as identified by Walczak M. et al. and Joias RM. et al. At the same time, Kacprzyk B. et al. showed that regardless of the number of remeltings, the same modifications occur in the alloy's composition, and the microstructural appearance does not show significant differences. The results on Co-based alloys support the findings of the authors mentioned earlier regarding the chemical composition of the alloy. [17] [18] [19]

With an increase in the number of remeltings and, possibly, variations in the proportion of recycled alloy (remelted), other separate phases may appear in the microstructure. The level of finishing and orientation of the dendritic structure obtained during the castings is systematically more advanced in the case of CoCrMoW alloy.

Besides inclusions, another indicator highlighted as responsible for the presence of residual stresses was oxides. Analyses performed on a sample taken exclusively from the remaining residues in the crucible after a number of four remeltings of the metal alloy showed, on one hand, an atypical sigma phase generated by the metastable stage of inclusions, and on the other hand, chromia and silicon dioxide - SiO_2 were identified alongside the oxides of chromium. This result led the authors to conclude that there is a high risk regarding the fracture of the prosthetic superstructure. [20]

Considering the results obtained in the conducted study and the data from the specialized literature, we emphasize the necessity of continuous and integrative research regarding the influence of using remelted alloys.

Chapter 4 - Comprehensive characterization of a biocompatible alloy from the Ti-Zr binary system for use in implant-prosthetic rehabilitation.

4.1. Introduction

The latest trends in implant-prosthetic rehabilitation highlight the necessity of assimilating new metallic materials that do not contain toxic elements and do not cause allergies. These materials should possess excellent mechanical and processing properties while also meeting the requirements for oral implantology use.

Over the last decade and a half, research has led to the development and implementation of binary applications based on Ti and Zr (Ti-Zr alloys) with highly promising biocompatibility and corrosion resistance, making them highly applicable in the medical field, including implant-prosthetic rehabilitation. They are utilized for manufacturing dental implants and prosthetic restorations.

4.2. Material și and method

The objective of obtaining and studying a Ti-Zr binary alloy, with approximately 20% Zr and the rest Ti (around 80%), originated from studying the ROXOLID alloy from the Straumann company. [21] The chosen approach involved analyzing the material's properties and observing whether adding a percentage higher than 18% Zr could improve the Ti-Zr system alloy through a series of experimental studies to characterize the resulting alloy composition, structure, and mechanical properties. The studies were conducted at the Faculty of Science and Engineering of Materials, University Politehnica of Bucharest.

The stages in this study were as follows:

Stage 1 - Obtaining the Ti-Zr binary alloy with controlled chemical composition of the raw materials.

Stage 2 - Characterization of the obtained alloy in terms of composition, structure (macrostructure, microstructure, and fine structure), and mechanical properties.

4.3. Results

Stage 1

The obtaining and processing of the Ti-20Zr ingot involved the preparation and use of precursors that were introduced into a crucible in the decreasing order of melting points, as required by the working protocol of the used furnace. Zirconium was introduced first, followed by titanium.

Characterization of the alloy was performed in terms of composition, structure (macrostructure, microstructure, and fine structure), and mechanical properties.

The aim of the study was to obtain information regarding the qualitative and quantitative analysis of the obtained alloy and its crystalline structure through X-ray diffraction.

For the characterization of the Ti-20Zr alloy, spectrometric, elemental, diffractometric, optical microscopy, tensile strength, and structural analyses were conducted.

4.4. Discussions

The properties of titanium alloys are superior to pure titanium, which leads to the desire to develop new materials that are optimal in terms of mechanical properties, non-toxicity, and non-allergenicity. Considering this need, the introduction of new elements that meet these requirements is sought, necessitating the elimination and replacement of elements such as vanadium (V), aluminum (Al), beryllium (Be), etc., which are harmful to the human body. [22] [23] [24] [25] [26]

It has been identified that a composition of 20% or 30% Zirconium (Zr) provides the alloy with excellent mechanical properties and an optimal biological response. Additionally, scientific research conducted by Ou P. et al. indicates that the Ti-20Zr alloy (in weight %) obtained through powder metallurgy exhibits excellent elasticity modulus (49.2 GPa) and outstanding osteointegration properties, making it an ideal alloy for dental implant applications. [27] Therefore, choosing the Ti-20Zr alloy to improve properties such as corrosion resistance, mechanical strength, and greatly enhanced biocompatibility is justified.

In the course of this research, a comparison was made between the properties obtained by applying thermo-mechanical treatment and the properties of the alloy in its cast state. The results of investigating the alloy's structure using X-ray diffraction have shown no indications of the presence of the β-phase or an intermediate phase. Due to Zr's larger atomic radius compared to Ti (1.62 and 1.47 Å, respectively), it causes an increase in the lattice parameters of the α-phase, resulting in the peaks shifting towards the lower angle. Given that the phases present in a Ti alloy depend on its chemical composition and cooling conditions during casting, rapid cooling rates have led to the formation of martensitic structures. These results are similar to those obtained by M. Takahashi et al. [28] [21]

Furthermore, the tensile testing of the two ingots, one in its cast state and the other treated thermo-mechanically, showed that the treatment significantly improved the properties, except for elongation, which decreased by three percentage points.

According to the literature, the elastic modulus of hard tissues falls within the range of 0.4-84.3, and the tensile strength ranges from 10-133 MPa, indicating that the investigated alloy exhibits superior mechanical properties compared to hard tissues. [29] [30]

The tensile strength of the untreated Ti-20Zr alloy is 589.21 MPa, while the thermo-mechanically treated alloy reaches 782.19 MPa. In comparison, the Ti-13-18Zr alloy has a tensile strength of 1000 MPa, and cortical bone tissue has values between 70-150 MPa. [29] [30] [21]

Regarding Young's modulus, the Ti-13-18Zr alloy has a value of 103.7 GPa, while the untreated Ti-20Zr alloy has 81.56 GPa, and the thermo-mechanically treated sample reaches 107.4 GPa, indicating a slight increase, about 4 units higher than the Ti-13-18Zr alloy. [21] [31]

In comparison to the Ti-13-18Zr alloy, which has a hardness value between 270-340 (310-320), the untreated Ti-20Zr alloy has a lower hardness. However, after deformation, the hardness increases and remains within the range specified in the literature for the Ti-13-18Zr alloy. [29] [21 Thermo-mechanical treatment improves the properties of the Ti-Zr binary alloy.

Chapter 5 - Statistical research regarding the materials and techniques used in implant-prosthetic treatments.

5.1. Introduction

The present chapter aims to evaluate the types of suprastructure commonly used by dentists in dental offices, the techniques applied in the implant-prosthetic treatment plan, as well as the level of quality of the works carried out in dental laboratories in relation to the established international practices. Additionally, this analysis involves correlating and interpreting the results obtained at the level of the sample used with official statistical data at the national and European levels. This approach aims to provide additional contextual information regarding the decisions made by dentists, considering the specific access of Romanian patients to dental medical services. The main objective of the research was to determine the aspects addressed by dentists regarding the selection of materials composing the superstructures of metal works, methods, the influence of materials on patients' health, the long-term resistance of superstructures and materials, biocompatibility, toxicity level, costs, etc. At the same time, the research will also take into account the correlation of the synthesized information from the responses of dentists in this survey with the practices employed by dental technicians in specialized laboratories.

5.2. Material și method

The research method used is a questionnaire, in which 67 dentists who practice in Romania participated, both in public and private healthcare settings in urban and rural areas. Out of the received responses, 12 questionnaires were invalidated due to incompleteness, while 55 were validated, statistically analyzed, and interpreted both quantitatively and qualitatively. The questionnaire was distributed in electronic format through Google Forms from January to December 2020. For the statistical analysis of the results, we used Jamovi software version 6.1 - Jamovi project 2021. Specifically, frequency analysis and the chi-square test of correspondence were applied to test for significant differences between the observed frequencies of participants' responses for each variable. [32] [33]

5.3. Results

Question no. 1: "Among the types of implant-supported superstructures used in partial edentulism, which variant/s do you frequently utilize? The question may have multiple answers."

The first question in the questionnaire aimed to identify the most commonly used techniques of implant-supported superstructures in partial edentulism. According to the results, the distribution of responses regarding the types of implant-supported superstructures in partial edentulism is as follows: the largest proportion of respondents, 33.3%, stated that they only use metal-ceramic superstructures. 29.8% declared that they use both metal-ceramic and zirconia superstructures, while the remaining 36.9% use both metal-acrylic, metal-composite, metal-ceramic, and zirconia (10.5% metal-acrylic, metal-composite, metal-ceramic, and zirconia - 8.8%, 7% metal-acrylic, metal-ceramic, and zirconia; 3.5% metal-acrylic and metal-ceramic, 1.8% metal-acrylic - metal-acrylic, and 1.8% metal-composite). The results of the chi-square test of correspondence indicate that the use of implant-supported superstructures with metal-ceramic and metal-ceramic and zirconia is significantly more frequent ($\chi2 = 62.33$, $p < .001$).

Question no. 2: "Which of the variables below do you consider in proposing the type of treatment through implant-supported superstructures?"

Regarding the variables considered in proposing the type of treatment through implant-supported superstructures, the frequency analysis indicates that the most important factors are the quality of the materials used (68.42%) and the cost of the work (21.05%).

Question no. 3: "What type of fixation for the implant-supported superstructure do you most commonly use?"

Regarding the type of fixation for the implant-supported superstructure, 63.8% use screw-retained fixation, and 36.2% use cement-retained fixation. The results of the chi-square test of correspondence indicate that screw-retained fixation is significantly more frequent ($\chi2 = 62.33$, $p < .001$).

Question no. 4: "Before choosing the type of metal-supported implant superstructure, do you perform specific metal allergy tests?"

According to the results, before choosing the type of metal-supported implant superstructure, 15.5% of respondents perform specific metal allergy tests, 1.7% perform tests only in case of allergy suspicion, and 82.8% do not apply specific allergy tests. The chi-square test of association indicates that the lack of application of specific metal allergy tests is significantly more frequent ($\chi2 = 68.22$, $p < .001$).

Question no. 5: "Before fixing the superstructure on the implant, do you perform specific cement allergy tests?"

Before fixing the superstructure on the implant, 6.9% of respondents perform specific cement allergy tests, 1.7% do not use cement, and 91.4% do not apply specific allergy tests for cement. The chi-square test of association indicates that the lack of application of specific cement allergy tests is significantly more frequent ($\chi2 = 87.42$, $p < .001$).

Question no. 8: "Does the technician you collaborate with inform you that they used recycled metals in casting?"

The question was formulated to highlight whether dentists are informed about the methods used by dental technicians in dental laboratories. For the item "Does the technician you collaborate with inform you that they used recycled metals in casting?", 15.5% of participants responded affirmatively, while 84.5% answered that they do not receive this information from the technician. The chi-square test of association indicates that the lack of information about the use of recycled metals is significantly more frequent ($\chi2 = 27.66$, $p < .001$).

Question no. 9: "Does your technician inform you about the method of producing metal-supported works?"

The purpose of question no. 9 was to identify communication channels between the dental technician and the dentist when discussing the production of metal-supported works. According to the results, 36.36% of participants responded that the technician they collaborate with informs them about the method of producing metal-supported works, while 62.1% of respondents do not receive information about the method of production. Specifically, the lack of knowledge about the method used by the technician for producing metal-supported works is more frequent ($\chi2 = 4.09$, $p = .046$).

5.4. Discussions

The choice of the type of suprastructure in oral rehabilitation with the help of implants is made by considering the quality of the materials used, but the next numerical frequency is related to costs.

In cases of oral rehabilitation, according to the responses to question no. 1, dentists prefer the use of metal-ceramics and zirconium among the most commonly used types of implant superstructures. According to the research by Burke et al., the metals present in implant superstructures are usually made from alloys based on Chromium-Nickel, Chromium-Cobalt, or precious metals such as gold, titanium, or platinum. [34]. Non-noble metal alloys based on Chromium-Nickel and Chromium-Cobalt are among the most commonly used in international practice due to their strength and affordable price, even though it is proven that Chromium-Nickel alloys can cause allergic reactions. [35]

Regarding the variables taken into account when the type of treatment with implant superstructures is proposed by the dentist, the responses also reveal the importance of the aesthetics criterion, a "sine qua non" component of dental medicine, which, among other things, has been defined as "the art and science of dentistry applied to create or enhance an individual's beauty within the limits of function and physiology." [36]

As for the type of fixation used most often for implant superstructures, the majority of responding dentists prefer the option of fixation by screwing. Residual cement can form through the cementation technique of crowns and can cause inflammation in the gingival mucosa, gingival recession, which can lead to the impairment of the peri-implant bone tissue, which, in the end, can compromise the inserted implant. [37]

Through question number 4 "Before choosing the type of superstructure on metal-supported implants, do you perform specific metal allergy tests?", the existence of preventive conduct that dentists should impose in the current practice of the dental office was proposed to be evaluated. It is necessary to have a special paraclinical protocol for this type of situation, considering that the problems raised by allergies are extremely diverse and are usually related to a multitude of conditions that can be triggered in satellite or distant organs. I consider it essential to return to one of the simplest principles of medicine, namely that every individual is unique, and obviously reacts uniquely to allergens, intolerances, or toxins. [38] The fact that 84.5% of dentists responded that they do not perform allergy tests for the metals used in implant superstructures practically affirms the theory that we rely on statistical precepts rather than on the particularities of each patient.

There is an intrinsic connection between the metals used in medicine and neurodegenerative diseases such as Parkinson's and Alzheimer's. [38] [39] Furthermore, when we discuss the metals and alloys used in the fabrication of suprastructures for oral rehabilitation with the help of implants, we also refer to the effects of electric currents that two metals present in the oral cavity can produce in larger or smaller quantities. [40] This reaction is called galvanism and generates electrochemical pollution which, in contact with saliva, a complex electrolyte, produces neurotoxic effects.

Due to the microelectric discharges, the maxillary nerve and the trigeminal nerve can be affected, and when affected, they can develop severe trigeminal neuralgia. [41] These vegetative disorders can lead to sleep disturbances, tinnitus, dizziness, inner ear disorders, and vertigo. [42]

Corrosion is another important reaction encountered. In oral surgery, titanium is a frequently used metal due to its biocompatibility with peri-implant tissue, optimal osseointegration, and corrosion resistance. However, when inserted in vivo, titanium is susceptible to generate corrosion reactions. Olmedo D.G. et al. conducted research from the specialized literature, which was later validated through laboratory studies. In the work "The issue of corrosion in dental implants: a review," they address the perspective of titanium implant rejection due to mechanical causes or titanium fatigue. However, laboratory studies revealed that the failure of the implant was actually attributed to the presence of corrosion. [43] Guglielmotti and Cabrini found metal particles in bone tissue and bone marrow. [44]

Regarding the practice of casting recasted metals (question 8), the data indicates that most dentists do not receive information regarding this aspect from the technicians they collaborate with.

The fabrication of metal-based prosthetic structures is done by alloying metals, respectively, through the conventional method of melting them using oxy-gas, induction, or with the help of additive and digital design methods through sintering or milling. [45]

Melting metal alloys is a crucial stage because the melting range must be optimal, i.e., the one recommended by the manufacturer, given that the casting process requires a relatively long time. The alloying of multiple metal compounds causes each metal to have a different melting point from the other alloyed metal. The melting range is essential to maintain the mechanical and chemical properties of the finished alloy and, most importantly, not to affect the patient's health. The fact that a prolonged melting range can create oxides may lead to contamination of the prosthetic structure. [46]

In the same measure, the materials used for melting and casting the prosthetic metal structure are essential. The crucibles used must also comply with certain standards, the quality of the alloys must be superior, and the procedures carried out for melting and casting the metal alloy must strictly follow the manufacturer's instructions, etc. [47] Essentially, clear and concise protocols are necessary. It is not by chance that certain aspects of the process of creating metal suprastructures have been mentioned. Scientific studies have investigated the incidence of procedures used in dental laboratories on various cases of cytotoxicity [48] [49], galvanism, contamination of alloys, and subsequent release of metal ions into the body [50] etc., and have concluded that the techniques used can bring significant changes at the cellular level. [51]

As a result of incorrectly applying technical procedures or repeatedly recycling metal alloys, alterations in the structure and microstructure of the alloys occur, leading to a finished product susceptible to structural and/or physical damage. Some of the consequences that may occur are related to microstructural changes. These changes can generate transformations that affect the mechanical-physical properties [52] and modifications in the dendritic structures compared to those commonly present in the matrix [53]. Another effect can be the alteration of the alloy's chemical composition [52]. Notable differences in porosity can be observed compared to the new alloy [54]. Residual inclusions and contamination with oxides can also be observed [46]. All these have been demonstrated through the studies conducted in this thesis and, according to the specialized literature, ultimately lead to cytotoxic effects on the human body. [16]

In question number 9, dentists were questioned whether they are informed about the method of fabricating metal-supported works by technicians. Here comes the question of whether dentists request the necessary details regarding the fabrication of works, but this aspect will probably be the subject of further research. It is essential to analyze the fact that there are many patients who complain of problems such as fracturing or cracking of the ceramics on metal-supported structures, which arise from the procedures used in fabricating metal-supported works.

From a technical point of view, there are three methods of fabricating metal structures - crowns, fixed partial prostheses, and other dental works - which are classified as follows:

1. The classic method of metal casting
2. The milling method
3. The sintering method

Chapter 6 – Conclusions and personal contributions

6.1. Conclusions

Study 1 - Studies on the influence of the casting process of alloys used in implant-prosthetic restorations on chemical composition and microstructural characteristics.

The results highlight two important aspects: 1) the use of a new crucible and a worn crucible influences the arrangement of the dendritic structure in the alloy and consequently, the properties of the alloys; 2) the use of remelted alloys in different mass percentages influences the microstructure, leading to the appearance of other independent phases that subsequently affect the mechanical properties. The greater the amount of remnant material in the alloy, the greater the influence of the presence of different elements such as Cr, Co, and W in the case of

Ni-Cr alloys, and Cr, Mo, W in Co-Cr alloys. In the case of Ni-Cr alloys, the Cr element tends to segregate at the surface of the melt, while Co and W tend to segregate in the mass of the melted alloy. In the case of Co-Cr alloys, the relatively different proportions of the intermetallic compound defined by the Co3Cr type are influenced by the preexistence of Cr, Mo, W elements (in the old crucible) and vary depending on the cooling rate and, implicitly, the degree of segregation of these elements.

Based on the results obtained for the studied alloys and the research in the specialized literature, it can be stated that there is no consensus protocol for the maximum number of remelting cycles, precisely due to the large number of influencing factors, such as the nature of the raw material, the obtaining method, the melting/remelting technique, the involved parameters, the type of crucible used, etc.

Second study - Comprehensive Characterization of a Potential Biocompatible Alloy from the Ti-Zr Binary System for Use in Implant-Prosthetic Rehabilitation.

Following specific laboratory analyses, the characteristics of the Ti-Zr alloy were determined, obtained through casting and melting in a levitation furnace at over 2000 °C. The resulting ingot was subjected to tests to estimate the elemental composition, structure, and mechanical properties. The verification of the chemical composition of the Ti-Zr alloy resulting from the casting technique was carried out by analyzing the estimation of the combined uncertainty together with the extended uncertainty, resulting in a minor coefficient of relative uncertainty, confirming the accurate measurement of the alloy's chemical composition. X-ray diffractometric analyses were also performed for two test samples, one of Ti and one of Ti-Zr. The results were similar for both samples, indicating that the Ti-Zr alloy exhibits the same characteristic lines as titanium, with minor modifications in the angles, which are irrelevant for the entire alloy. The microstructural analysis of the cast Ti-Zr alloy revealed that the obtained micrographs show a compact hexagonal martensitic structure throughout the volume and a well-defined Widmanstatten structure with dendritic blocks.

Following the analysis of the structurally deformed state of the Ti-Zr alloy through thermo-mechanical processing, an average dimensional ratio of 1:8 was observed. The results of the tensile strength and microhardness tests highlighted that, as a result of thermo-mechanical processing, the Ti-Zr alloy possesses superior properties.

The use of two distinct bioelements, such as zirconium and titanium, can replace other elements such as vanadium or aluminum, metals which, according to scientific studies, can produce long-term toxic and allergic reactions. The Ti-Zr binary nature confers corrosion resistance, which means that saliva maintains a normal pH and does not become acidic to favor an electrolytic, inflammatory environment in the oral cavity. According to clinical analyses conducted by the Swiss manufacturer Straumann, for the Roxolid implant made from 85%Ti-13-18%Zr, the healing process is more advanced than for implants made from other titanium alloys. The implant's strength is also considerably increased, with better cytocompatibility than pure titanium. Therefore, we can conclude that the Ti-20%Zr alloy is a perfect substitute for the classic titanium alloy, Ti-Zr, bringing significant improvements.

Third study – Statistical Research on Materials and Techniques Used in Implant-Prosthetic Treatments.

It is essential to develop standardized procedures between dentists and dental technicians to reduce the fabrication time of prosthetic works. Especially the sharing of information regarding the metal alloys used, their origin - whether through recycling, using the same crucible, etc. - are crucial aspects that can determine the choice of the most appropriate prosthetic solution tailored to the patient's clinical situation and, of course, their needs.

Certainly, a higher degree of awareness on both sides regarding the effects of different metals used in prosthetic works would increase the number of toxicological and metal allergy tests performed. This is because most dentists do not conduct tests to assess allergic reactions to metals or cements.

6.2. Personal contributions

1. Conducting a study on the use of new and reused crucibles, as well as combinations of new and re casted alloys, which demonstrated the increased susceptibility of Ni-Cr alloys to contamination with residues of Cu, Co, and Fe already present in the remnant material (in the case of reused crucibles) and the better stability of Co-Cr alloys compared to Ni-Cr alloys regarding contamination with remnant elements. (Chapter 3)
2. Conducting studies that highlighted the possibility of obtaining binary Ti-based alloys by replacing Al and V with Zr, leading to improved mechanical properties of the alloy. (Chapter 4)

3. Conducting research on the techniques, methods, and materials used in suprastructures for oral rehabilitation with the help of implants. (Chapter 5)

6.3. Future research directions

1. Expanding the studies on contamination and its effects on the physical, chemical, and biocompatibility properties of Ni-Cr and Co-Cr alloys.
2. Investigating the relationship between the reuse of instruments and compositional changes in other types of alloys.
3. In vivo testing of the Ti-20Zr alloy obtained.
4. Identifying the need for implementing an informational flow regarding the services offered by dental technicians.
5. Demonstrating the level of cytotoxicity of the Cr-Ni alloy and studying the link between the reuse of instruments and compositional changes in other types of alloys.
6. Another research direction could involve testing the cytotoxicity level of dental materials that contain beryllium oxides, an extremely hazardous chemical element for human health, which is present in metal alloys such as chromium, nickel, iron, used for creating prosthetic suprastructures.

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