DOI: https://doi.org/10.2298/SARH190706112S
UDC: 615.46:616.314-7

ORIGINAL ARTICLE / ОРИГИНАЛНИ РАД

Selective laser melting and sintering technique of the cobalt-chromium dental alloy

Dejan Stamenković[1], Kosovka Obradović-Đuričić[1], Rebeka Rudolf[2], Rajko Bobovnik[3], Dragoslav Stamenković[4]

[1]University of Belgrade, School of Dental Medicine, Belgrade, Serbia;
[2]University of Maribor, Faculty of Mechanical Engineering, Maribor, Slovenia;
[3]Faculty of Polymers Technology, Slovenj Gradec, Slovenia;
[4]Serbian Medical Society, Academy of Medical Sciences, Belgrade, Serbia

SUMMARY
Introduction/Objective The objective of this paper is to describe the microstructure and mechanical properties of sintered Co-Cr alloy and to emphasize its advantages and disadvantages with respect to the microstructure and mechanical properties of cast Co-Cr alloy.
Methods Base Co-Cr alloy, EOSint M EOS Co-Cr SP2 (EOS GmbH, Munch, Germany), was used for the purpose of this research as the base material for sintering metal structures of metal-ceramic restorations. Metal sintering was conducted by using EOSint M 280 device of German origin in a stream of neutral gas – argon. After that, the alloy was heated over a period of 20 minutes at the temperature of 800°C. The chemical composition of the alloy was determined by energy dispersive spectroscopy. Microstructure of the tested alloy samples was examined under an optical metallographic and scanning electron microscope. Physical and mechanical properties were measured in a universal testing machine. The samples were prepared according to the standard ISO 527-1:1993.
Results Chemical composition of the sintered Co-Cr alloy, determined by applying energy dispersive spectroscopy, indicated the same qualitative but different quantitative composition compared to cast Co-Cr alloys. The microstructure of the sintered Co-Cr alloy is lamellar in nature, with two dominant phases: ξ-Co and/or ξ-Cr (fcc – *face-centered cubic*) and γ-Co (hcp – *hexagonal close-packed*). Mechanical properties of the Co-Cr alloy obtained by applying selective laser melting technology compared to the cast Co-Cr alloy are superior or approximately the same.
Conclusion Selective laser melting of the Co-Cr alloy is a good example of new technologies based on digitization. Together with other digitized procedures, this technology is an introduction to a new era in dentistry popularly called Dentistry 4.0. The advantages of the selective laser melting technology with respect to the conventional technology of casting Co-Cr alloy metal structures are precise metal structure fitting and eco-friendly technology.
Keywords: selective laser melting; sintering technique; Co-Cr alloy

INTRODUCTION

Dental alloys represent a very dynamic field of dentistry. Changes that occur in this area, in fact, reflect the developments of basic scientific technologies. Mechanical and biological properties of the same alloy are largely dependent on the technological processes of forming the alloy into dental restorations. The process of alloy melting and casting for dental purposes has been known for centuries and melting and casting conditions have been constantly improved – from primitive alloy melting by applying naked flame and open-air casting, to melting by applying induced current in vacuum or neutral gases. Nevertheless, even the perfect casts have certain flaws.

A completely new approach to forming dental restorations appeared with the third, and soon after, with the fourth industrial revolution. The third industrial revolution, also known in the field of dentistry as Digital Dentistry or Dentistry 3.0, introduced numerous new procedures based on digital technologies (3D imaging, intraoral scans, computer-aided design and computer-aided manufacturing – CAD-CAM, cone-beam computer tomography – CBCT, computer-aided implantology). The transition from the third to the fourth industrial revolution, i.e. to Dentistry 4.0, was barely noticeable. Dentistry 4.0 is not a completely new technology. In this case new system solutions were created on the platform (infrastructure) originating from the previous digital revolution. This revolution introduced greater automation in dental laboratory procedures, i.e. diagnostic and therapeutic procedures in dental offices. The selective laser melting (SLM) and compacting (sintering) of metal powder particles is a step forward in the modern dental practice. This technology plunged us into the fourth industrial revolution, i.e. Dentistry 4.0 [1, 2, 3].

The process of making dental restorations by sintering dental alloys basically includes three steps: digital impression, designing virtual restoration, and 3D printing [4–7].

Digital impression, suitable for further computer processing, can be obtained by direct 3D

Received • Примљено: July 6, 2019
Revised • Ревизија: October 7, 2019
Accepted • Прихваћено: October 8, 2019
Online first: October 28, 2019

Correspondence to:
Dejan STAMENKOVIĆ
23 Deligradska Str.
Belgrade 11000
Serbia
dr.dejan.stamenkovic@gmail.com

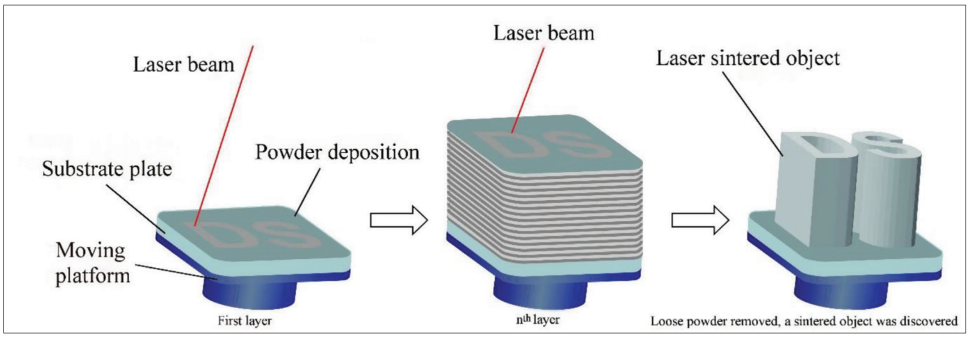

Figure 1. Outline of schematic functioning principles of selective laser melting [7]

digitization in the patient's mouth (intraoral scanners) and indirect 3D digitization of the gypsum model (extraoral scanners) [7, 8]. Nowadays, these scanners, in addition to precision, ensure great comfort for both the doctor and the patient.

Designing virtual restoration, i.e. computerized modeling framework of crown, bridge or removable partial denture, represents the second step in advanced technology of making dental restorations. Virtual restorations are designed by using commercial computer packages, which are computer tools that facilitate and speed up the design process [9]. Virtual restoration files (STL files) are sent directly to the software of the machines designed to make metal frameworks of dental restorations. At this stage, one intermediate step is also possible. If the design control is required in real space, obtained STL files are sent to 3D printers, which print out a model of the future fixed or mobile dental prostheses in polymer or, less often, wax. The detected defects can still be remedied.

The third step is laser melting of the Co-Cr alloy powder and producing the metal framework by sintering. In the process of selective laser sintering, the object is printed by successive addition of thin, horizontal layers. Each layer is printed by applying a thin layer of alloy powder over previously made object, which is then melted with a laser beam in the form of the following layer [10, 11, 12].

Upon cooling, the melted metal powder is bonded horizontally (thus forming a new layer) and vertically (bonding with previously made layer). The form of each layer is determined by a computer, based on a virtual restoration model (obtained STL files). The process of powder melting is governed by cross-sections determined in such manner. The process of sintering only ensures the bond of the laser melted powder (Figure 1) [7].

The objective of this paper is to describe the microstructure and mechanical properties of the sintered Co-Cr alloy and to emphasize its advantages and disadvantages with respect to the microstructure and mechanical properties of the cast Co-Cr alloy.

METHODS

Base Co-Cr alloy, EOSint M EOS Co-Cr SP2 (EOS GmbH, Munch, Germany), was used for the purpose of this research as the base material for sintering metal structures of metal-ceramic restorations. Metal sintering was conducted by using EOSint M 280 device of German origin in a stream of neutral gas – argon. After that, the alloy was thermally treated over a period of 20 minutes at the temperature of 800°C.

The chemical composition of the alloy was determined by energy dispersive spectroscopy (EDS analysis). Microstructure of the tested samples was examined under an optical metallographic microscope (MM) and scanning electron microscope (SEM) in the Materials Testing Laboratory at the Faculty of Mechanical Engineering in Maribor, Slovenia. Physical and mechanical properties were measured in a universal testing machine in the Materials Testing Laboratory at the Faculty of Polymer Technology, Slovenj Gradec, Slovenia. Six samples were prepared according to the ISO standard 527-1:1993.

RESULTS

Chemical composition of the sintered Co-Cr alloy, determined by applying EDS, indicated the same qualitative composition as for cast Co-Cr alloys. However, there were certain differences in the quantitative composition of the alloy, with the values for W, Si, and O being higher (Figure 2, Table 1).

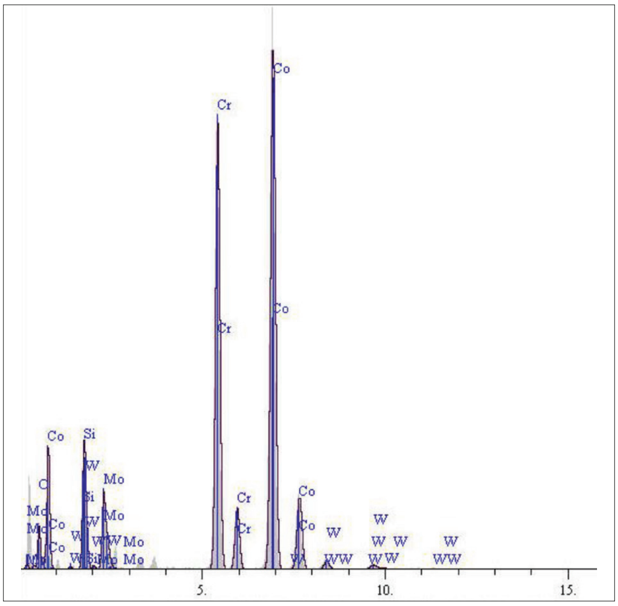

Figure 2. Results of the energy dispersive spectroscopy of EOS Co-Cr SP2 alloy after sintering and thermal treating

Table 1. Numerical values of elements of energy dispersive spectroscopy of EOS Co-Cr SP2 alloy after sintering and thermal treating

Elt.	Line	Intensity (c/s)	Error 2-sig	Atomic %	Conc.
O	Ka	22.40	0.947	8.878	2.578 wt%
Si	Ka	43.85	1.324	4.954	2.342 wt%
Cr	Ka	404.78	4.024	27.583	26.932 wt%
Co	Ka	511.87	4.525	54.323	58.110 wt%
Mo	La	52.34	1.447	2.813	4.899 wt%
W	Ma	43.78	1.323	1.810	6.038 wt%
			100.000	100.000	wt%

Table 2. Test results for physical and mechanical properties of the selective laser melting (SLM) builds

Samples – SLM		Samples – SLM + thermal treatments	
Tensile strength	800 MPa	Tensile strength	900 MPa
0.2% yield strength	600 MPa	0.2% yield strength	700 MPa
Elongation	10%	Elongation	2%
Modulus of elasticity	170 GPa	Modulus of elasticity	180 GPa

The composition and conditions for compacting particles (sintering) determine the alloy structure. Sintered Co-Cr alloy is examined under MM (Figure 3) and SEM (Figure 4).

Microporosity and porosity, i.e. the presence of dendrites due to contraction, are characteristic for cast Co-Cr alloys. Microscopic examinations of the sintered Co-Cr alloy showed slightly more homogeneous and slightly more porous structure compared to the cast Co-Cr alloy. (Figure 5).

Mechanical properties of the sintered Co-Cr alloy, prior to thermal treating, indicate that the tubes are significantly more brittle compared to the cast Co-Cr alloy. However, after thermal treating, physical and mechanical properties are approximately the same or superior (Figure 6, Table 2). Figure 7 shows the SEM micrograph of the fractured tube surface after mechanical testing.

The roughness of the metal surface is significant, both for the bond between the metal and cement, and for the bond between the metal and ceramics. The roughness of the metal surface concerned ensures better strength of both bonds. SEM micrograph of sintered Co-Cr alloys in this study shows uniform roughness (Figure 8).

DISCUSSION

The results obtained are in accordance with relevant data found in the literature referring to the chemical composition of EOS Co-Cr SP2 alloy determined based on EDS analysis, but also based on X-ray diffractometry analysis (XRD) performed by other authors [13, 14]. Chemical compositions of alloys differ slightly depending on the manufacturer and the surface that the analysis was performed on.

The microstructure of the sintered Co-Cr alloy is lamellar in nature, with two dominant phases: ε-Co and/or ε-Cr (fcc – face-centered cubic) and γ-Co (hcp – hexagonal close-packed). This structure was determined based on XRD analysis [15, 16, 17]. The microstructure of two types of samples is observed: a sintered sample and a sintered and thermally treated sample. The same structure with slightly lower intensity of peaks is determined with the thermally treated sample [17].

Microstructure of the sintered Co-Cr alloy does not indicate intermetallic phases, contrary to the cast Co-Cr alloy. Upon casting, Co-Cr alloys create an intermetallic phase (Cr_7C_3 and $Cr_{23}C_6$) [15]. In theory, the structures obtained by applying SLM technology are not porous. However, this should be taken with some reserve, since the porosity of sintered structures depends on the purity of the input components (alloy powder) and sintering conditions (environment, temperature). The alloys without intermetallic phases and with minimum porosity have better mechanical properties [13]. A very precise, homogeneous alloy with good mechanical properties is obtained by laying one layer of the alloy powder over another, as confirmed by various authors (Meacock and Vilar [18], Castillo-Oyagüe et al. [19]).

Mechanical properties of the Co-Cr alloy obtained by applying the SLM technology, which are most commonly described, are the following: properties determined based on stress–strain diagram and the metal-ceramic bond strength. The main purpose of metal sintering is to obtain the metal with the highest possible density [20]. Metal density depends on the temperature of the thermally treated metal and the amount of energy required for melting metal powder on one side and scanning, laser power, and the thickness of the powder layer and the thermally treated region on the other.

Jevremović et al. [21] and Zhou et al. [22] demonstrated that sintered and thermally treated Co-Cr alloys show a significantly higher tensile strength and greater modulus of elasticity than cast Co-Cr alloys. Unlike these authors, Lu et al. [23] demonstrated that the density, hardness, and electrochemical properties of the compensation do not depend on the technique applied and that both types meet the requirements of the ISO 22764:2006 standard.

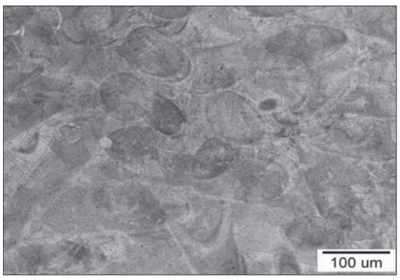

Figure 3. Metallographic microscope micrograph of the sintered Co-Cr alloy surface

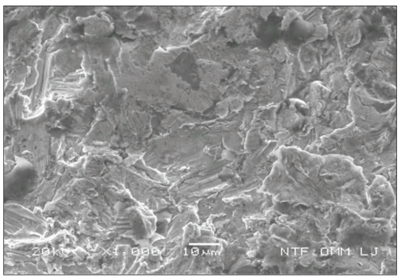

Figure 4. Scanning electron microscope micrograph of the sintered and thermally treated Co-Cr alloy surface

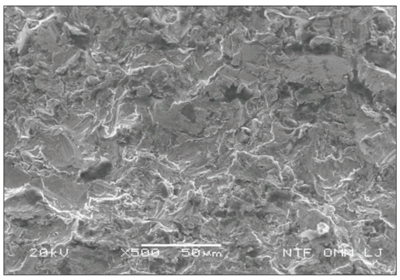

Figure 5. Scanning electron microscope micrograph of the sintered, thermally treated, and sandblasted Co-Cr alloy surface

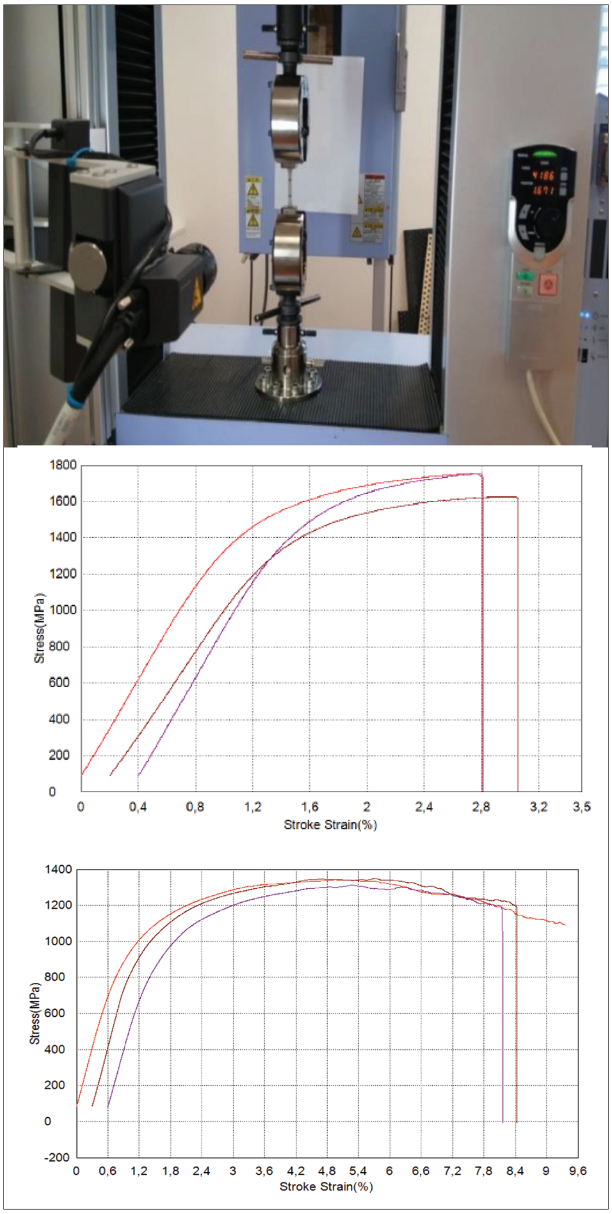

Figure 6. Universal testing machine (A) and stress–strain diagrams for the Co-Cr alloy after sintering (B) and after sintering and thermal treating at 800°C (C)

Residual stress appears as a result of thermally treated individual layers of the melted metal powder. Quick heating is accompanied by quick cooling, which leads to metal expansion, followed by the shrinkage of the metal. This is most striking immediately after the removal of the alloy from the machine, and it is remedied by releasing the residual stress, i.e. by thermal treatment of the alloy. For the purpose of our research, thermal treatment (releasing residual stress) is conducted in the furnace, first at the temperature of 450°C (45 minutes) and then at the temperature of 750°C (60 minutes). After the expiration of the 60-minute period, the furnace is turned off, and the furnace door is opened at the temperature of 600°C, only to turn off the stream of protective gas (argon) at the temperature of 300°C.

Sintered Co-Cr alloy shows higher hardness compared to the same cast alloy. Relevant data found in the literature indicate that the hardness of sintered dental Co-Cr alloys ranges 440–475 HV10, i.e. 382 HV10, whereas the hardness of the cast Co-Cr alloy ranges 325–374 HV10 [24, 25, 26]. Higher hardness and more homogeneous microstructure result in increased corrosion and wear resistance [24]. Subsequent thermal treating of the sintered alloy during the process of baking ceramics (in case of metal-ceramic restorations) does not affect its corrosion resistance [26].

Relevant data found in the literature indicate that the average surface roughness (the profile roughness parameter) immediately after sintering is about 8 μm [27]. After sand-blasting Al_2O_3, the roughness is reduced due to surface homogenization and uniformization. The roughness of the sintered Co-Cr alloy surfaces is several times greater than the roughness of the cast alloy surfaces. This may cause a problem when making mobile restorations (e.g. removable partial denture framework). On the other hand, a rough surface increases the wettability and reduces the contact angle, which enhances the bond between the metal and the ceramics [28].

The SLM technology for the Co-Cr alloy, as a piece in a mosaic, perfectly fits into the technological process automation in smart dental laboratories. This technology uses cyber-physical systems, the Internet, and cloud computing as its platform. In combination with diagnostic information (3D imaging, intraoral scans, etc.) and treatment plan, digital impression and simulation in a virtual articulator, the SLM technology represents a major step towards automation in patient diagnostics and therapy, a major step towards the fourth industrial revolution – Dentistry 4.0. In addition to the already described advantages of sintered alloys in terms of their microstructure and physical and mechanical properties, this technology also ensures significant time saving (dentist's time, patient's time, lab time).

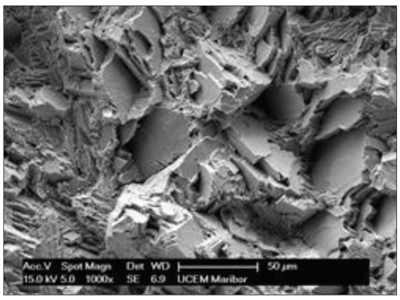
Figure 7. Scanning electron microscope micrograph of a fractured sintered Co-Cr alloy tube surface after mechanical testing

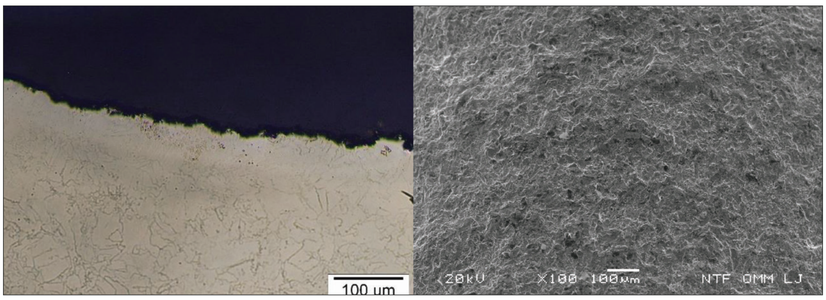

Figure 8. View of the surface (A) and cross-section (B) through the edge of the selective laser melting sinter Co-Cr alloy

Time is money, thereby meaning cheaper diagnostics and therapy. Another significant advantage of the SLM technology lies in the fact that it is an eco-friendly technology (smaller quantities of medical and other waste).

CONCLUSION

Selective laser melting of the Co-Cr alloy is a good example of new technologies based on digitization. Together with other digitized procedures (digital impression, designing virtual restoration, 3D printing), this technology is leading us towards Dentistry 4.0.

1. The qualitative composition of sintered Co-Cr alloys is the same as cast Co-Cr alloys. However, there are certain differences in the quantitative composition of the alloys (higher values for W, Si, and O in the sintered Co-Cr alloys).
2. The microstructure of the sintered Co-Cr alloy is lamellar in nature, with two dominant phases: ε-Co and/or ε-Cr (fcc – face-centered cubic) and γ-Co (hcp – hexagonal close-packed).
3. Mechanical properties of the sintered Co-Cr alloy, prior to thermal treatment, indicate that tested specimens are significantly more brittle compared to the cast Co-Cr alloy. However, after thermal treatments, physical and mechanical properties are approximately the same or superior.
4. The SLM technology has the following advantages over the conventional technology of casting Co-Cr alloy structures: precise metal framework fitting; digital impression and designing virtual restoration, which ensure avoiding mistakes that can occur due to shrinkage of the impression material, the expansion of the plaster that the working model is made of, the expansion of the refractory cast and the shrinkage of the casting upon cooling; eco-friendly technology.

Conflict of interest: None declared.

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Селективно ласерско топљење и синтеровање денталне легуре кобалт-хром

Дејан Стаменковић[1], Косовка Обрадовић-Ђуричић[1], Ребека Рудолф[2], Рајко Бобовник[3], Драгослав Стаменковић[4]

[1]Универзитет у Београду, Стоматолошки факултет, Београд, Србија;
[2]Универзитет у Марибору, Машински факултет, Београд, Србија;
[3]Факултет за технологију полимера, Словењ Градец, Словенија;
[4]Српско лекарско друштво, Академија медицинских наука, Београд, Србија

САЖЕТАК

Увод/Циљ Циљ рада је описати микроструктуру и механичке карактеристике синтероване легуре *Co-Cr* и истаћи њене предности и мане у односу на микроструктуру и механичке карактеристике ливене легуре *Co-Cr*.

Методе У истраживању је коришћена базна легура *Co-Cr, Eosint M EOS Co-Cr SP2* (*EOS GmbH*, Минхен, Немачка) за синтеровање металних конструкција металокерамичких надокнада. Синтеровање метала је обављено на апарату *EOSint M* 280 у струји неутралног гаса аргона. Након тога легура је жарена 20 минута на температури од 800°*C*. Хемијски састав легуре одређиван је енергодисперзивном спектроскопијом. Микроструктура испитиваних узорака легуре посматрана је на оптичком металографском и електронском скенирајућем микроскопу. Физичко-механичке карактеристике мерене су на универзалној кидалици. Узорци су припремани према стандарду *ISO* 527-1:1993.

Резултати Хемијски састав узорака синтероване легуре *Co-Cr* показао је исти квалитативан али различит квантитативан састав у односу на легуре *Co-Cr* за ливење. Микроструктура синтероване легуре *Co-Cr* је ламеларне природе, у којој доминирају две фазе: ε-*Co* и/или ε-*Cr* (*fcc – face-centred cubic*) и γ-*Co* (*hcp – hexagonal close-packed*). У поређењу са ливеном легуром *Co-Cr*, механичке карактеристике синтероване легуре *Co-Cr* су боље или приближно исте.

Закључак Селективно ласерско топљење легуре *Co-Cr* је добар пример нових технологија заснованих на дигитализацији. Заједно са другим дигитализованим процедурама које претходе, ова технологија је предворје новој ери у стоматологији, популарно названој *Dentistry* 4.0. Предности технологије селективног ласерског топљења у односу на технологију конвенционалног ливења металних конструкција од легуре *Co-Cr* су прецизност належања металне конструкције и чиста технологија.

Кључне речи: селективно ласерско топљење; синтеровање метала; легура *Co-Cr*