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**Paper Accepted\***

**ISSN Online 2406-0895**

**Original Article / Оригинални рад**

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

Селективно ласерско топљење и синтеровање  
денталне легуре кобалт-хром

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**Received: July 6, 2019**

**Revised: October 7, 2019**

**Accepted: October 8, 2019**

**Online First: October 28, 2019**

**DOI: <https://doi.org/10.2298/SARH190706112S>**

\***Accepted papers** are articles in press that have gone through due peer review process and have been accepted for publication by the Editorial Board of the *Serbian Archives of Medicine*. They have not yet been copy-edited and/or formatted in the publication house style, and the text may be changed before the final publication.

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When the final article is assigned to volumes/issues of the journal, the Article in Press version will be removed and the final version will appear in the associated published volumes/issues of the journal. The date the article was made available online first will be carried over.

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

### Селективно ласерско топљење и синтеровање денталне легуре кобалт-хром

#### 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 in the 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. Samples were prepared according to the standard ISO 527-1:1993.

**Results** Chemical composition of sintered Co-Cr alloy, determined by applying energy dispersive spectroscopy, has indicated the same qualitative but different quantitative composition as for cast Co-Cr alloys. The microstructure of the sintered Co-Cr alloy is of a lamellar nature, with two dominant phases:  $\epsilon$ -Co and/or  $\epsilon$ -Cr (fcc – face-centered cubic) and  $\gamma$ -Co (hcp – hexagonal close-packed). Mechanical properties of Co-Cr alloy obtained by applying selective laser melting technology compared to the cast Co-Cr alloy are better or about the same.

**Conclusion** Selective laser melting of Co-Cr alloy is a good example of new technologies based on digitization. Together with other digitized procedures this technology is introduction to a new era in dentistry popularly called - Dentistry 4.0. The advantages of 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

#### 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.

**Метод** У истраживању је коришћена базна легура Co-Cr, Eosint M EOS Co-Cr SP2 (EOS GmbH, Минхен, Немачка) за синтеровање металних конструкција метало-керамичких 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 енерго-дисперзивном спектроскопијом. Микроструктура испитиваних узорка 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 узорка синтероване легуре Co-Cr показао је исти квалитативан али различити квантитативан састав у односу на легуре Co-Cr за ливење. Микроструктура синтероване легуре Co-Cr је ламеларне природе у којој доминирају две фазе:  $\epsilon$ -Co/или  $\epsilon$ -Cr (fcc – face-centred cubic) и  $\gamma$ -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 налегања металне конструкције и чиста технологија.

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

## 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 (*3-D 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) metal powder particles is a step forward in modern dental practice. This technology plunged into the fourth industrial revolution, i.e. into 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 digitization in the patient's mouth (intraoral scanners) and indirect 3D digitization of 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 which 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, archived STL files are sent to 3D printers which print a model of the future fixed or mobile dental prostheses in polymer or, less often, in wax. The detected defects can still be remedied.

The third step is laser melting of Co-Cr alloy powder and making a 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 (archived 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 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 thermally treated in the 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. Physical and mechanical properties were measured in a universal testing machine in the Materials Testing Laboratory at the Faculty of Polymer Technology, Slovenj Gradec. Six samples were prepared according to the ISO standard 527-1:1993.

## RESULTS

Chemical composition of the sintered Co-Cr alloy, determined by applying energy dispersive spectroscopy (EDS – *Energy Dispersive Spectrometer*), has indicated the same qualitative composition as for cast Co-Cr alloys. However, there are certain differences in the quantitative composition of the alloy, namely: the values for W, Si, and O are higher (Figure 2, Table 1).

The composition and conditions for compacting particles (sintering) determine the alloy structure. Sinter Co-Cr alloy is examined under optical (metallographic – 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 have 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 thermally treated, indicate that the tubes are significantly more brittle compared to cast Co-Cr alloy. However, after thermally treated, physical and mechanical properties are approximately the same or better (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 - *X-ray diffractometry*) 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 of a lamellar nature, with two dominant phases:  $\epsilon$ -Co and/or  $\epsilon$ -Cr (fcc – *face-centered cubic*) and  $\gamma$ -Co (hcp – *hexagonal close-packed*). This structure has been determined based on XRD analysis [15-18]. 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 [18].

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 ( $\text{Cr}_7\text{C}_3$  and  $\text{Cr}_{23}\text{C}_6$ ) [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 et al. [19], Castillo-Oyagüe et al. [20]).

Mechanical properties of Co-Cr alloy obtained by applying 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 [21]. 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, the thickness of the powder layer and the thermally treated region.

Jevremovic et al. [22] and Yanan et al. [23] 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, Yanjin et al. [24] 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 standards ISO 22764:2006.

Residual stress appears as a result of thermally treated individual layers of melted metal powder. Quick heating is accompanied by quick cooling, which leads to metal expansion, followed by the shrinkage of 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 min) and then at the temperature of 750°C (60 min). After the expiry 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 from 440 to 475 HV<sub>10</sub> [25, 26], i.e. 382 HV<sub>10</sub> [27], whereas the hardness of cast Co-Cr alloy ranges from 325 to 374 HV<sub>10</sub> [26, 27, 28]. Higher hardness and more homogeneous microstructure result in increased corrosion and wear resistance [25, 26]. Subsequent thermally treated of the sintered alloy during the process of baking ceramics (in case of metal-ceramic restorations) does not affect its corrosion resistance [28].

Relevant data found in the literature indicate that the average surface roughness (the profile roughness parameter) immediately after sintering is about 8.0 µm [29]. After sand-blasting Al<sub>2</sub>O<sub>3</sub>, the roughness is reduced due to surface homogenization and uniformization. The roughness of sintered Co-Cr alloy surfaces is several times greater than the roughness of cast alloy surfaces. This may cause a problem when making mobile restorations (e.g., RPD framework). On the other hand, a rough surface increases the wettability and reduces the contact angle, which enhances the bond between the metal and ceramics [30].

SLM technology for 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 the cloud computing as its platform. In combination with diagnostic information (3-D imaging, intraoral scans, etc.) and treatment plan, digital

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

## CONCLUSION

Selective laser melting of 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 to the 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 alloy (higher values for W, Si, and O).
2. The microstructure of the sintered Co-Cr alloy is of a lamellar nature, with two dominant phases:  $\epsilon$ -Co and/or  $\epsilon$ -Cr (fcc – *face-centered cubic*) and  $\gamma$ -Co (hcp – *hexagonal close-packed*).
3. Mechanical properties of the sintered Co-Cr alloy, prior to thermal treating, indicate that tested specimens are significantly more brittle compared to cast Co-Cr alloy. However, after thermal treatments, physical and mechanical properties are approximately the same or better.
4. The advantages of SLM technology with respect to the conventional technology of casting Co-Cr alloy metal structures are the following:

Precise metal framework fitting. Digital impression and designing virtual restoration, 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; SLM technology is an eco-friendly technology.

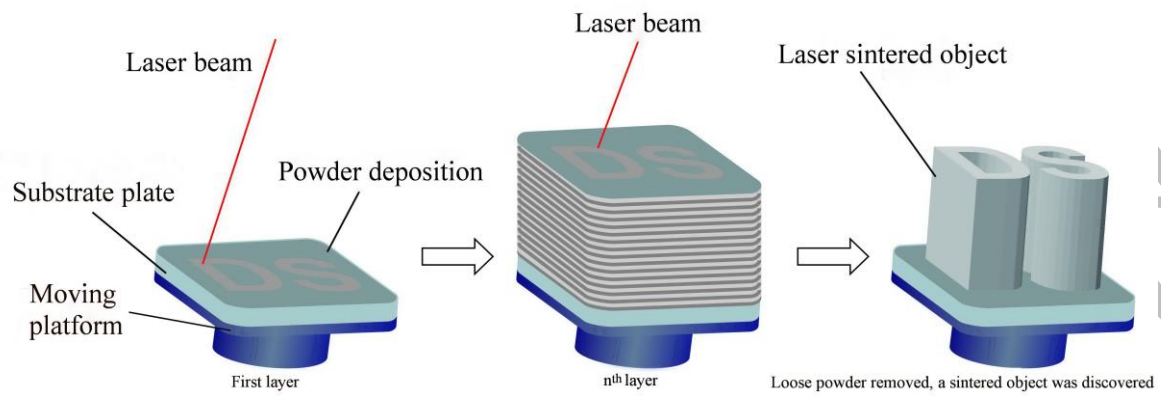
**Conflict of interest:** None declared.

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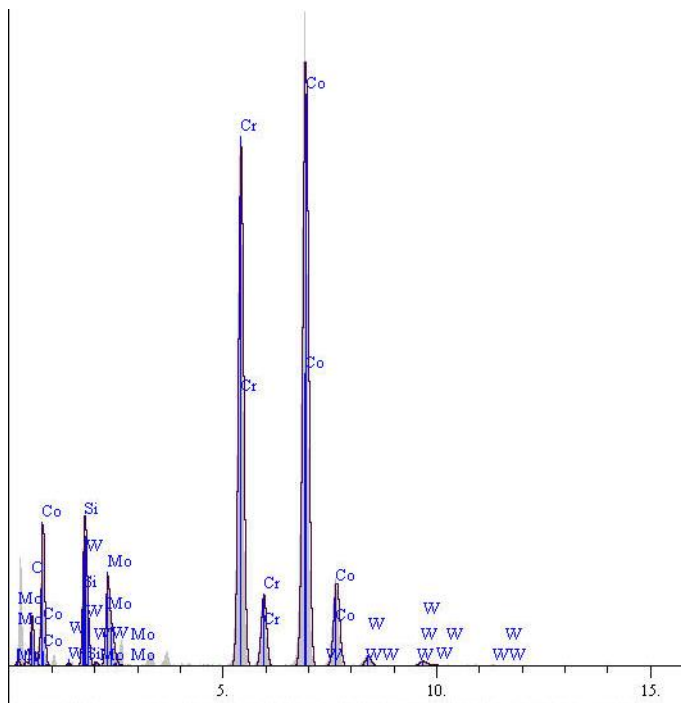
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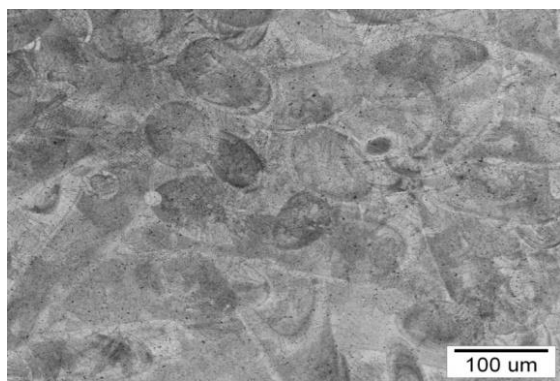
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**Figure 1.** Outline of schematic functioning principles of selective laser melting [7]

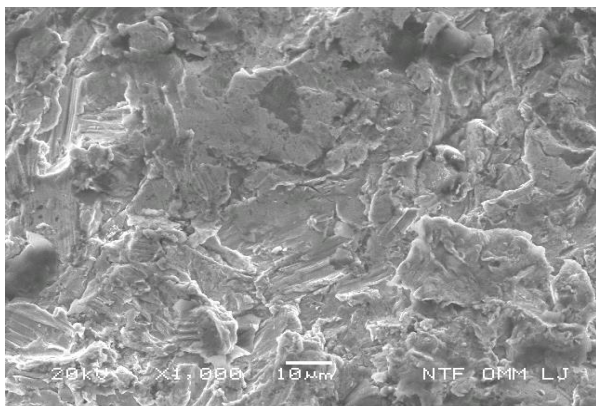


**Figure 2.** Results of the energy dispersive spectroscopy of EOS Co-Cr SP2 alloy after sintering and thermal treating



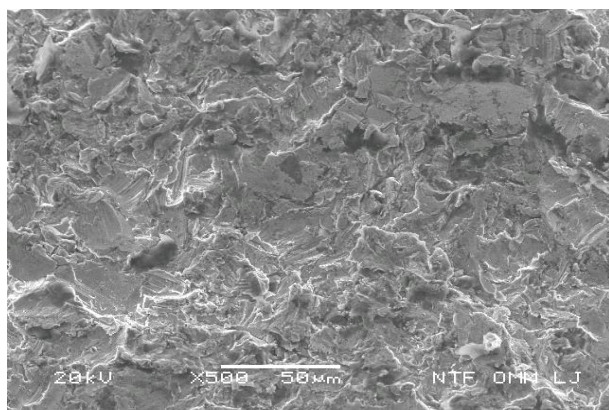
**Figure 3.** MM micrograph of the sintered Co-Cr alloy surface

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

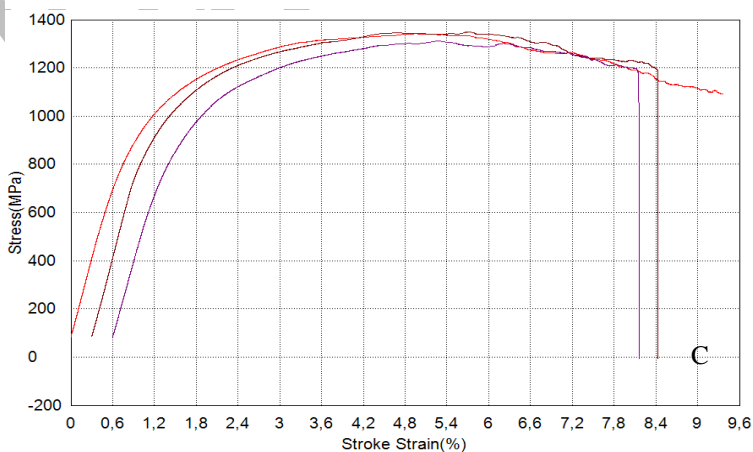
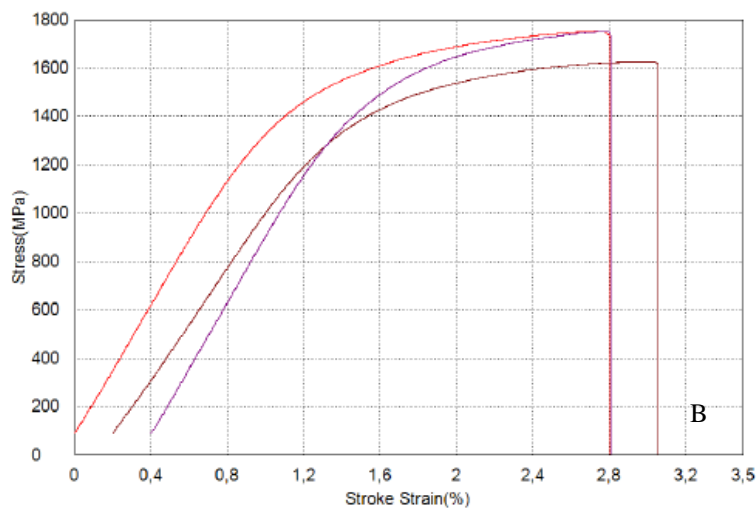
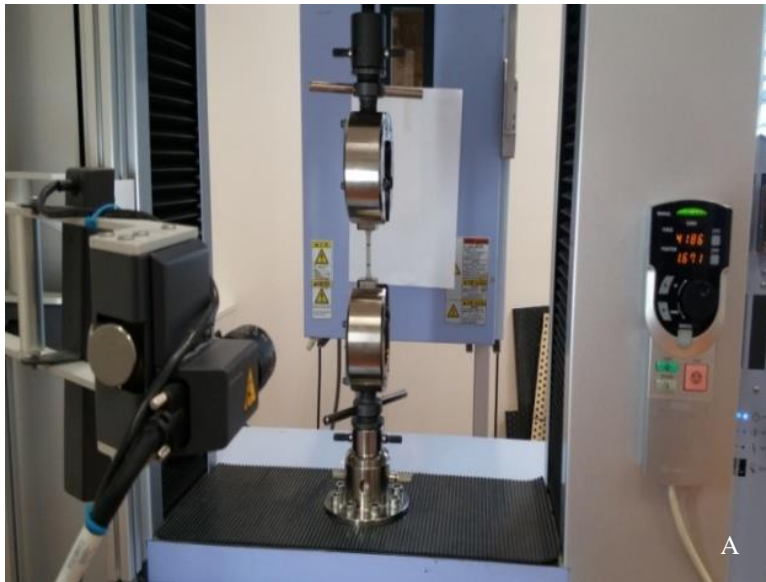
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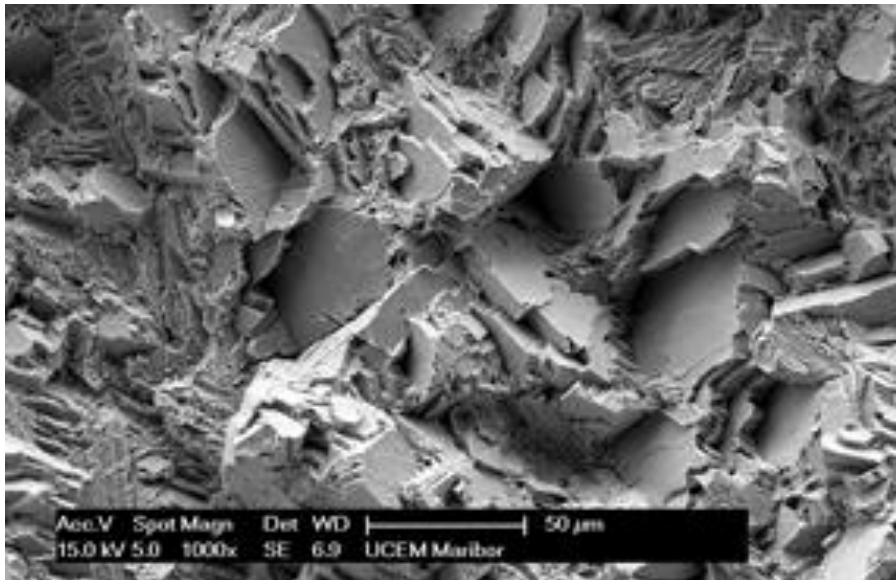
**Figure 5.** Scanning electron microscope micrograph of sintered, thermally treated and sandblasted Co-Cr alloy surface

Paper accepted

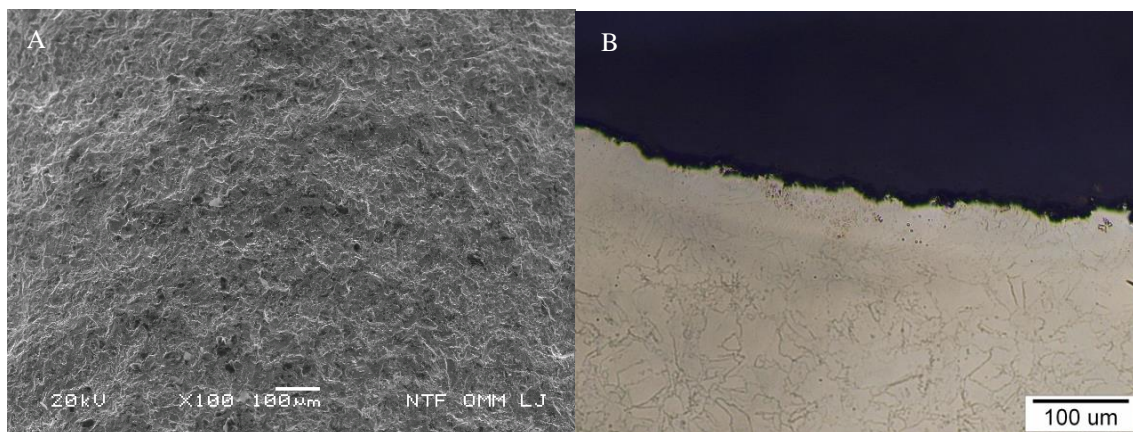




**Figure 6.** Universal testing machine (A) and stress-strain diagrams for Co-Cr alloy after sintering (B) and after sintering and thermal treatment at 800°C (C)



**Figure 7.** Scanning electron microscope micrograph of 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

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

<b>Elt.</b>	<b>Line</b>	<b>Intensity (c/s)</b>	<b>Error 2-sig</b>	<b>Atomic %</b>	<b>Conc.</b>
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%

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

<b>Samples – SLM</b>		<b>Samples – SLM + thermal treatments</b>	
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

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