

Surface texture and hardness of dental alloys processed by alternative technologies

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Abstract. Technological developments have led to the implementation of novel digitalized manufacturing methods for the production of metallic structures in prosthetic dentistry. These technologies can be classified as based on subtractive manufacturing, assisted by computer-aided design/computer-aided manufacturing (CAD/CAM) systems, or on additive manufacturing (AM), such as the recently developed laser-based methods. The aim of the study was to assess the surface texture and hardness of metallic structures for dental restorations obtained by alternative technologies: conventional casting (CST), computerized milling (MIL), AM power bed fusion methods, respective selective laser melting (SLM) and selective laser sintering (SLS). For the experimental analyses metallic specimens made of Co-Cr dental alloys were prepared as indicated by the manufacturers. The specimen structure at the macro level was observed by an optical microscope and micro-hardness was measured in all substrates. Metallic frameworks obtained by AM are characterized by increased hardness, depending also on the surface processing. The formation of microstructural defects can be better controlled and avoided during SLM and MIL process. Application of power bed fusion techniques, like SLS and SLM, is currently a challenge in dental alloys processing.

1. Introduction

Alternative manufacturing methods of dental restorations using base metal dental alloys are of ongoing interest, having a growing impact in the field of dental technology.

Technological developments have led to the implementation of novel digitalized manufacturing methods for the production of metallic structures in prosthetic dentistry [1-5]. The potential for fabricating metallic dental components with a complex geometry directly from digital data using automated equipment and appropriate materials is very significant. These technologies can be classified as based on subtractive manufacturing, such as the milling of premanufactured materials assisted by computer-aided design/computer-aided manufacturing (CAD/CAM) systems [2,5-7], or on additive manufacturing (AM), such as the recently developed laser-based methods. Although CAD/CAM has long been directly associated with the milling procedure in dental literature, it should be mentioned that AM procedures are also classified as CAD/CAM technologies.



Understanding of structural and microstructural defects present in metallic frameworks processed through AM techniques and post-processing protocols such as heat treatment to reduce these defects, are important in order to transform the microstructures to those acceptable for practice [8].

Selective laser melting (SLM) and Selective laser sintering (SLS) belong to Powder bed fusion, and consist in a thermal energy that selectively fuses regions of a powder bed [9]. Direct metal printing methods can generally be categorized as laser-based, electron beam-based, arc-based, and ultrasonic welding-based. Laser-based metal AM methods are classified into laser sintering (SLS), laser melting (SLM), and laser metal deposition (LMD) [10].

SLS is a powder bed fusion technique in which a scanning laser is used to consolidate sequentially deposited layers of a metal powder [11]. Different types of lasers including CO₂, disk, Nd:YAG, and fiber lasers are used. The principal consolidation mechanism is liquid-phase sintering involving partial melting and coalescence of the powder. SLM is a second powder bed fusion technique involving the consolidation of metal powders using powerful lasers. While the equipment setup and configuration and processing methodology are similar in SLS and SLM, in SLM, the powder is completely or nearly completely melted to produce a fully dense or nearly fully dense structure. SLM thus produces metal articles with a higher level of microstructural homogeneity compared with SLS.

2. Objective

The aim of the study was to assess the surface texture and hardness of metallic structures for dental restorations obtained by alternative technologies: conventional casting, computerized milling, AM power bed fusion methods, respective SLS and SLM.

3. Materials and methods

For the experimental analyses metallic specimens made of Co-Cr dental alloys were prepared using traditional casting (CST), computerized milling (MIL), selective laser sintering (SLS) and selective laser melting (SLM), as indicated by the manufacturers. Round plates of 20 mm diameter and 2 mm thick were fabricated using different technologies. The cast alloy was manufactured by conventional lost wax technique with a phosphate-bonded investment. The mold was cast with Wirobond® SG alloy (Bego, Bremen, Germany) at 900°C using a vacuum pressure casting machines Nautilus (Bego, Bremen, Germany). The mold was left to cool down to room temperature and the specimens were then divested, sandblasted with alumina particles (200 µm) and finished with rotative instruments, burs suitable for Co-Cr alloys. Type 4 alloy Wirobond® SG fulfils all criteria of the ISO 22674 and ISO 9693-1 Standards. The composition is Co 63.8; Cr 24.8; W 5.3; Mo 5.1; other constituents Si, and Fe. Wirobond® SG allows normal cooling and provides effective, economic processing in the laboratory.

A prefabricated block of a commercial Co-Cr dental alloy CopraBond K (White Peaks Dental Systems GmbH & Co. KG, Essen, Germany), a non precious blank on Co-Cr base, type 4, was milled to fabricate the sample using Datron D5 5-axis dental milling machine (Datron, Mühlthal, Germany). A disc wax pattern was digitized and the specimens were cut to their final dimensions. The composition is: Co 61.0, Cr 28.0, W 8.5, Mn 0.25, Fe <0.5, Si 1.65, C <0.1. CopraBond K is a nickel- and beryllium free Co-Cr blank, specially designed for CAD/CAM applications.

The laser-sintered and laser-melted specimens were prepared from commercial Co-Cr powder Starbond CoS Powder 16 (S & S Scheftner GmbH, Mainz, Germany) using a dental laser sintering device (PXS Dental System, Phenix Systems, Clermont-Ferrand, France), respective a SLM device MYSINT 100 (Sisma, Piovene, Italy). For scanning the high accuracy D700 scanner (3Shape, Copenhagen, Denmark) and for design Dental System™ CAD Software (3Shape, Copenhagen, Denmark) was used. The alloy is a type 4 alloy according to ISO 22674. Free of beryllium, nickel and cadmium. Nominal values of alloy composition in mass percent: Co 59.0; Cr 25.0; W 9.5; Mo 3.5; Si max. 1; other constituents: C, Fe, Mn, N max. 1.5.

The high power laser beam fused the powder granules homogeneously distributed on a flat building plate guided by a pre-determined pattern by computer aided design (CAD). The laser power, scanning speed, and the distance between two scanning line sand layers were the principal parameters in the

laser sintering and melting processes. These were set according to two recommendations for dental prostheses, respective metal processing, and using fibre laser for power bed fusion. Relief-firing was conducted under argon by heating up to 450°C within 60 minutes, holding for 45 minutes. The specimens resulted after CAD/CAM technologies were not additionally prepared.

Oxide-firing (at 950 – 980°C) was performed, the metal surface was sandblast again with fresh aluminium oxide (approx. 150 µm). All substrates were ground until 2000-grit SiC paper, polished with universal polishing paste (Ivoclar Vivadent AG, Schaan, Principality of Liechtenstein). They were after cleaned in alcohol, rinsed in distilled water and dried with adsorbent paper towels.

The specimen structure at the macro level was observed by an optical microscope Leica DM500 (Leica Microsystems, Wetzlar, Germany) using a reflective mode. Microstructural defects were measured using an image processing program, Image J.

Micro-hardness was measured in all substrates using Mitutoyo SJ 201 device (Mitutoyo Corporation, Kanagawa, Japan). The hardness value was obtained as a mean value of at least 5 indents for samples polished and sandblast with fresh aluminium oxide of approx. 75 µm and approx. 200 µm).

4. Results

Microstructural defects were observed by optical microscopy on well-polished surfaces of Cr-Co-Mo alloy specimens prepared by SLS, SLM, MIL, CST (Fig. 1).

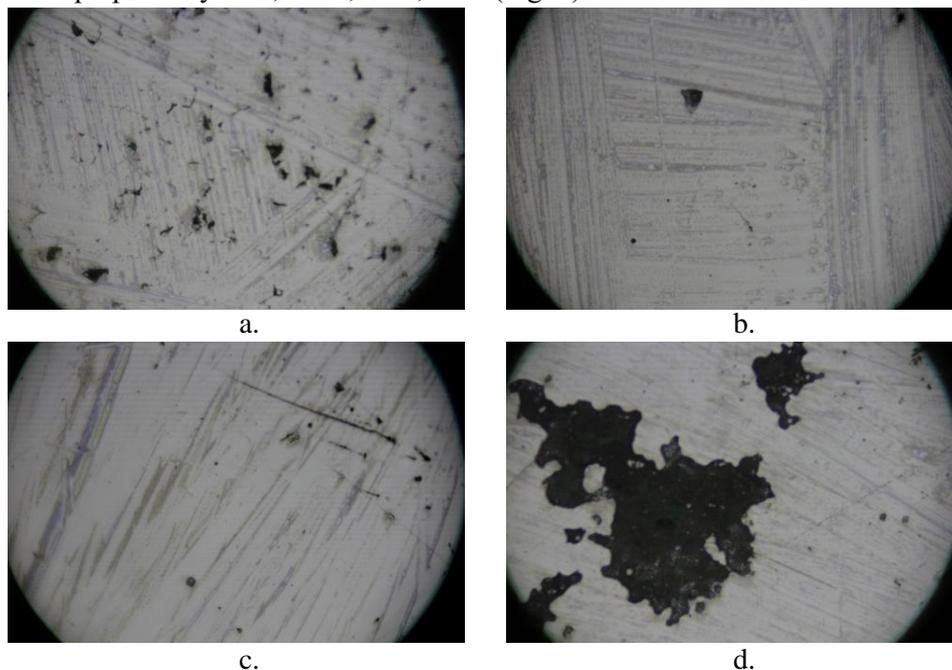


Figure 1. Microstructural defects observed by optical microscopy on well-polished surfaces of Cr-Co-Mo alloy specimens obtained by: a. SLS, b. SLM, c. MIL, d. CST.

Regarding microstructure the best results were obtained for SLM and MIL samples. Isolated or interconnected voids originating from hard agglomerates or insufficient packing of powder granules prior to sintering with sizes up to 45 µm were observed inside the specimens prepared by SLS. The surface of the specimens fabricated by CST contains grooved holes as a result of incomplete melting, with dimensions between 20 and 350 µm. In MIL samples traces were identified which were probably caused during the end-mill process and rare small voids (size up to 20 µm). SLM obtained specimens revealed the best microstructure, with the fewest voids, with dimensions up to 25 µm.

The hardness values recorded for specimens sandblasted with aluminium oxide of 75 µm were the highest for all samples, followed by those sandblasted with aluminium oxide of 200 µm and lastly from the polished (Table 1). Thus the preparation of the frameworks for ceramic veneering using

aluminium oxide of 75 μm is founded also from this point of view. With respect to the manufacturing technique, AM obtained specimens were associated with the highest HV mean values, about two times higher than those processed by MIL and CST .

Table 1. Vickers hardness mean value of the Co-Cr-Mo alloy specimens prepared by SLS, SLM, MIL, CST.

| Samples | polished | sandblasted aluminium oxide 75 μm | sandblasted aluminium oxide 200 μm |
|---------|----------|--|---|
| SLS | 431 | 509 | 452 |
| SLM | 478 | 506 | 474 |
| MIL | 230 | 277 | 273 |
| CST | 215 | 233 | 231 |

5. Discussions

Given the large differences in the manufacturing process between casting, which uses the complete melting and overheating of casting materials, the milling of a prefabricated metal block and AM of a fine metallic powder, large differences in microstructural characteristics and hardness values can be anticipated [12].

AM allows obtaining of complex geometries more than subtractive methods. The application of the AM methods, recently introduced in dental technology, and studies using the clinical implications are necessary. By identifying differences in processing methods and material characteristics, future studies should look up for reducing disadvantages [13, 14].

Surface texture metrology can be used as a means of gaining insight into the physical phenomena taking place during the manufacturing process and process variables, through examination of the surface features generated by the process. It becomes a powerful exploration tool, increasing knowledge of the process and ultimately allowing the creation of improved manufacturing processes [15-17]. Surface texture is the geometrical irregularities present at a surface. Surface texture does not include those geometrical irregularities contributing to the form or shape of the surface [18]. The diversity of AM processes as well as the large number of key process parameters that change from build to build makes the use of traditional means of process qualification less than satisfactory [19]. These new challenges require a more profound understanding of the AM technology and process, and will ultimately require the development of AM surface texture good practice guidance, specifications and standards [20].

6. Conclusions

1. The potential for fabricating metallic dental components directly from digital data represents an opportunity.
2. Metallic frameworks obtained by AM are characterized by increased hardness, depending also on the surface processing.
3. The formation of microstructural defects can be better controlled and avoided during SLM and MIL process.
4. Application of power bed fusion techniques, like SLS and SLM, is currently a challenge in dental alloys processing.

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