THE INFLUENCE OF PROSTHETIC ELEMENTS MANUFACTURING TECHNOLOGY ON PROPERTIES AND MICROSTRUCTURE SHAPING Co-Cr-Mo ALLOYS

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Abstract

The presented publication discusses the test results regarding samples of a prosthetic alloy from the Co-Cr-Mo system. The test samples were obtained by means of two different methods applied in prosthetics laboratories to compare their properties and microstructure. To obtain the samples via the traditional lost wax casting method, the cast alloy Co-Cr-Mo was used, commercially known as Wironit LA. In the case of the modern technique DMLS (Direct Metal Laser Sintering), metallic powder Co-Cr-Mo, called EOS Cobalt Chrome MP1, was used. The samples of both Co-Cr-Mo alloys obtained via the two methods were prepared for metallographic tests; they also underwent microstructural observations with the use of light microscopy (LM) and scanning electron microscopy (SEM), and next they were subjected to hardness tests. The obtained samples demonstrated a dendritic structure. In the samples cast with the lost wax casting method, a segregation of the chemical composition was revealed. The samples obtained by means of the DLMS method were characterized by chemical composition homogeneity. The hardness measurements with the statistical analysis of the measurement results showed a difference between the examined alloys. On the basis of the performed studies, it was stated that the applied methods of manufacturing prosthetic elements make it possible to obtain diversified microstructural and mechanical properties of the alloys. The hardness value significantly affects the subsequent mechanical and finishing treatment of prosthetic elements, such as metal bases of crown caps, bridges, mobile prostheses or other retention elements.

Keywords: *Co-Cr-Mo alloys, dental prosthetics, lost wax casting method, selective laser sintering of metal powders (DLMS), microstructure, hardness*

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Introduction

The modern techniques applied to recreate toothing losses have to meet growing requirements. The development of dental prosthetics provides more and more possibilities to select the optimal method of filling losses in the patient's toothing [1-3]. A group of metallic materials commonly applied in prosthetics are Co-Cr alloys with Mo and/or W microadditions [4-7]. The technology of producing metal prosthetic elements determines their microstructure [6,8-11,32], thus affecting their mechanical properties [12], corrosion resistance [13] and tribological wear [14,15,27].

One of the most popular methods used to manufacture prosthetic elements is the lost wax casting [16-18] which makes it possible to match the element with the patient's anatomy. Another frequently applied method is direct metal laser sintering (DMLS) [16,17,19]. Thanks to these methods elements of complicated shapes can be manufactured, hence the high interest in their possibilities in dental prosthetics.

The production of a metal prosthetic element via the precision casting method (lost wax technique) takes place during the casting process. In this process, a previously prepared ceramic mould based on a wax model is filled with a liquid metal alloy. The quality of the obtained casts depends largely on the metal's melting point among other factors [20,21]. Already at the beginning of the modelling process, special attention should be paid to a uniform thickness of the prosthetic element's wall [23]. In the lost wax casting method, in its first stage, a wax model representing the shape of the final prosthetic element is produced. The wax moulds are placed in a casting ring and covered with a protective body, consisting of a binding agent, such as calcium sulphate hemihydrate ($CaSO_4*2H_2O$) and a refractory ceramic mass of quartz or cristobalite. The casting ring is left for about 45 min until the ceramic mass completely binds. The process of wax burning consists in heating the casting ring in order to harden the refractory mass and removing the wax by melting and burning. If during the ceramic mass cooling, a thermal expansion occurs, the wax burning temperature is within the scope of 500-600°C. In turn, if a hygroscopic expansion takes place during the ceramic mass cooling process, the wax burning temperature should be maintained at about 460°C [20,23]. Directly after the wax burning process, the casting process should begin so that the casting ring does not cool down significantly [23]. The liquid alloy (Co-Cr-Mo) from the temperature of 1350-1400°C is cast into a hot mould with a temperature of 800-1000°C. In order to uniformly distribute the metal in the casting mould, a centrifuge with a centrifugal force is used. In this method, the metal is introduced from the crucible where it has been melted, directly through the casting channels, into the casting mould located in the ring. The whole process in the centrifuge does not last longer than 1 second. After the casting process, the centrifuge should continue to exert pressure on the solidifying metal, as this guarantees the proper shaping of the cast edges, and the mould has to cool down. During that time, the alloy crystallization takes place. Next, the ceramic mould is broken, and the metal prosthetic element is cleaned, milled and polished [20].

It should be emphasized that producing a high-quality cast is possible only if the recommendations are met and the appropriate melting and casting temperatures for a given alloy are applied. If the parameters are improper, the cast metal element will have an inhomogeneous structure and lowered mechanical properties. Additionally, casting defects, such as pores and micro shrinkages, will occur.

In consequence, the lost wax casting makes it impossible to produce a precision cast with efficient microstructural and mechanical properties. That is why an increasing role in prosthetics laboratories is played by DMLS (Direct Metal Laser Sintering) techniques which belong to the most popular additive technologies (additive manufacturing) [24]. The laser does not sinter but completely melts the powdered material. Therefore, the obtained elements are very strong and suitable for the subsequent treatment and their structure is not weakened [25]. The dimensional accuracy grade of the DMLS techniques according to DIN EN ISO 2768 is category C, depending on the size of the detail (in practice from +/-0.15 for details of up to 100 mm to +/-0.3 for larger details, depending on the geometry type and the arrangement in the working chamber). In the series production of one detail, the process can be optimized and it is possible to obtain accuracies of more than +/-0.1 mm. The minimal wall thickness of metal prints equals 0.6 mm [26]. In the additive techniques, numerically controlled devices apply the CAD/CAM (Computer-Aided Design/Computer-Aided Manufacturing) technology. CAD systems are used to design the shape of crowns and bridges, whereas CAM makes it possible to generate a machine code based on the created CAD models.

CAM packets also visualize and simulate the production process so as to limit errors. The virtually designed components are free of imperfections that might occur in the lost wax method [20,21,27,28].

The aim of the performed research was to evaluate the effect of the two production methods, i.e. the lost wax casting method to produce a prosthetic alloy Co-Cr-Mo and the direct metal laser sintering method (DMLS) to produce metallic powder Co-Cr-Mo, on the obtained material microstructure and hardness.

Materials and Methods

The materials assigned for the tests were the Co-Cr-Mo alloys samples used to produce prosthetic elements, such as crowns, bridges, attachments and frameworks of partial dentures. Some of the test samples were obtained through remelting of the commercial alloy Wironit LA by Bego Company [29] during the lost wax casting process. The chemical composition and the mechanical properties of the alloy Co-Cr-Mo Wironit LA are presented in TABLES 1 and 2.

TABLE 1. Chemical composition of the tested Co-Cr-Mo alloy (Wironit LA), % wt. [29].

Cr	Mo	Si	C	Co
29.0	5.5	1.2	max. 0.25	rest

TABLE 2. Physical and mechanical properties of Co-Cr-Mo alloy (Wironit LA) [29].

ρ [g/cm^3]	Melting temp. [°C]	Casting temp. [°C]	E [GPa]	$R_{p0,2}$ [MPa]	R_m [MPa]	A_5 [%]
8.2	1300-1340	1450	220	640	940	8

In the lost wax method, soft modelling wax was used to make models of elements measuring 3x19x19 mm which casting pins were fixed to. The construction was fixed to the base of the casting ring with a pin. The prepared protective mass was poured into the casting ring and left for 20 min until a complete solidification of the mould took place. In the following step, the ceramic mould was placed in the furnace heated up to 970°C in order to melt the wax. Next, the hot ceramic mould and the crucible with the Co-Cr-Mo (Wironit LA alloy) were placed in the centrifuge equipped with an inductive heater. The centrifuge was activated and the Co-Cr-Mo liquid alloy was pressed into the ceramic mould due to the centrifugal force. The temperature of the liquid alloy was about 1400°C. After the casting process, the mould was removed and cooled in the air until the complete alloy crystallization. At the last stage, the cast Co-Cr-Mo alloy samples were cleaned of the remains of the ceramic mass with a sandblasting unit and an abrasive disk. Next, the channels and the casting cone were cut off. A total of 10 Co-Cr-Mo samples measuring 3x19x19 mm were cast and subjected to tests.

The second batch of the test materials were the Co-Cr-Mo samples obtained via the direct laser metal sintering technique (DLMS). The EOS CobaltChrome MP1 powder was used to prepare the samples. It fulfils the chemical and mechanical specifications of the standards ISO 5832-4 and ASTM F75 for casting alloys Co-Cr-Mo assigned for implants, as well as the specifications of the standards ISO 5832-12 and ASTM F1537 for alloys Co-Cr-Mo assigned for forged elements. The chemical composition and the mechanical properties of the powder EOS CobaltChrome MP1 are presented in TABLES 3 and 4 [30].

The laser sintering process was performed with a 3D EOSINT M280 printer by means of the DMLS technique. The initial value of the laser beam equalled 1 mW, and the laser wavelength was 600-700 nm. Nitrogen was applied as a protective gas. The detail made on the printer was shaped as a cylinder measuring 140 mm in length and 10 mm in diameter. The shape and size of the produced element required the use of a support mesh to protect the element from bending during the printing, as the powder layer beneath the element did not withstand the pressure. The experiments were performed on a total of 10 Co-Cr-Mo samples measuring 10 mm in length and 10 mm in diameter.

TABLE 3. Chemical composition of the tested Co-Cr-Mo powder (EOS CobaltChrome MP1), % wt. [30].

Cr	Mo	Si	C	Mn	Fe	Co
26-30	5-7	≤ 1.0	≤ 0.16	≤ 1.0	≤ 0.75	rest

TABLE 4. Physical and mechanical properties of Co-Cr-Mo powder (EOS CobaltChrome MP1) [30].

ρ [g/cm^3]	$R_{p0,2}$ [MPa]	R_m [MPa]	A_5 [%]
8.3	600	1100	20

The metallographic tests of the examined Co-Cr-Mo alloys included the samples grinding and polishing in order to obtain a flat surface and their etching to reveal the microstructure. Abrasive papers (SiC) with the granularity 320, 500, 800, 1000, 1200 and 2000 μm were used for grinding and polishing cloth with an agent of a diamond suspension 3 and 1 μm was applied for polishing. The finishing treatment was conducted on a polishing cloth type OPS with the use of a diamond suspension ¼ μm. In order to reveal the microstructure of the examined samples, the microsections were subjected to electrolytic etching. The etching reagent was a mixture of 60 cm^3 HCl, 15 cm^3 HNO_3, 15 cm^3 CH_3COOH and 15 cm^3 distilled water and the voltage used for the electrolytic process was 4-5 V. The microstructure observations were performed with a light microscope (LEICA DM4000 M) and a scanning electron microscope (HITACHI S-3500N) equipped with an X-ray spectrometer EDS by NORAN 986B-1SPS.

The samples' hardness was measured by means of the Vickers method using a hardness tester ZWICK/ZHU 187.5, with a load of 100 N (HV10) and a measurement time of 10 s. The tests were conducted on all the 20 samples obtained by both production techniques and the number of measurements in randomly selected areas was 10. The obtained measurement results were subjected to statistical analysis. The following parameters were determined: the hardness measurement mean value ($\overline{HV10}$), the standard deviation ($s_{(\overline{HV10})}$), the absolute limiting error (δ_g) and the relative limiting error (γ_g), where tα = 1.98 - the value read from the t-Student distribution table, dependent on the number of performed measurements, the significance level α = 0.05 and the number of degrees of freedom k = 9 (k = n - 1, where n constitutes the number of measurements performed on the sample).

Results and Discussion

The etched Co-Cr-Mo alloys samples underwent observations by means of light microscopy. The selected micro-images of the examined alloys, both after casting by the lost wax method and after laser sintering by DLMS, are presented in FIGs 1 and 2.

The Co-Cr-Mo (Wironit LA) alloy samples (FIG. 1a-d) are characterized by the dendritic microstructure, typical of casting alloys. It is visible especially under low magnification (FIG. 1a). Based on the literature data [31], we know that the alloy's primary structure obtained directly after crystallization consists of large matrix grains constituted by a solution of chromium and molybdenum in cobalt. As a result of the structural and thermal stresses present in the material, microcracks occurred, causing a drop in the material's strength. The crack propagation proceeded in the interdendritic spaces (FIG. 3a,b).

FIG. 4a shows the typical microstructure of the cast Co-Cr-Mo alloy obtained by lost-wax casting with visible precipitates enriched in Mo and Cr. It is known that cast materials are characterized by the chemical composition micro-segregation and inhomogeneity. That is why, the SEM observations and EDS chemical composition analyses were carried out in micro-areas (FIG. 4b,c).

The chemical composition analyses performed in micro-areas of the lost-wax cast Co-Cr-Mo alloy confirmed that its matrix was constituted by a solid solution of chromium and molybdenum in cobalt (FIG. 4c). The SEM observations and the analysis of X-ray spectra revealed the phenomenon of inverse Cr micro-segregation (FIG. 3 and 4) which diffused both into the dendritic cores and the interdendritic spaces.

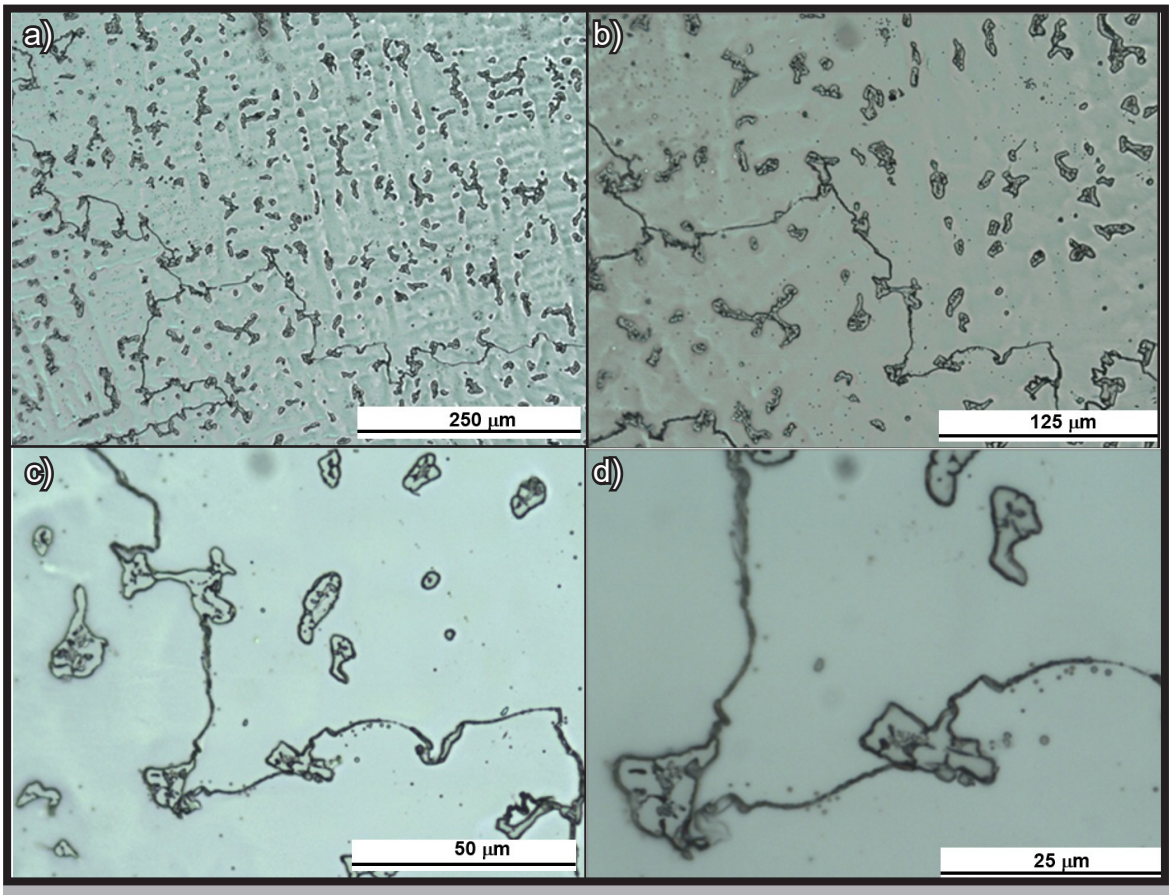

FIG. 1. Microstructure of Co-Cr-Mo alloy after the lost-wax casting method (a-d); LM, etched state.

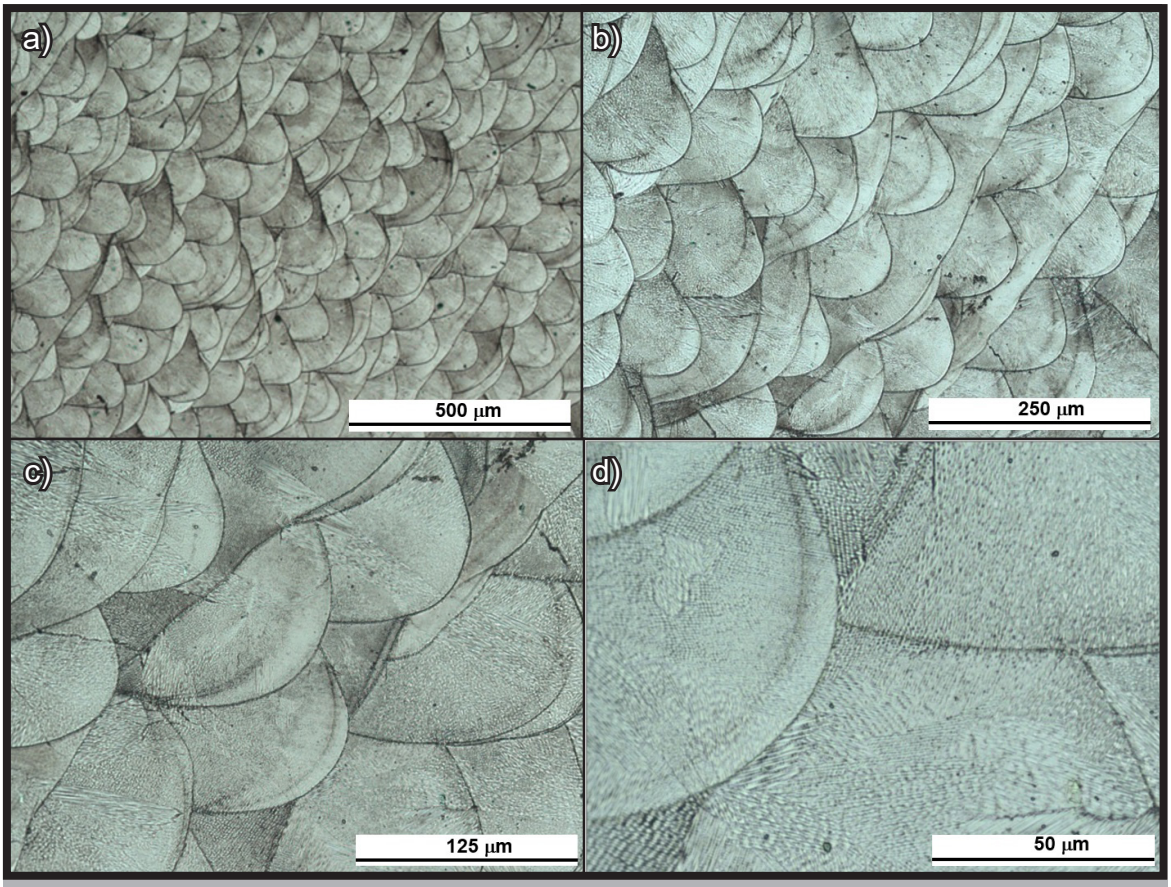

FIG. 2. Microstructure of Co-Cr-Mo alloy after selective laser sintering using the DLMS technique (a-d); LM, etched state.

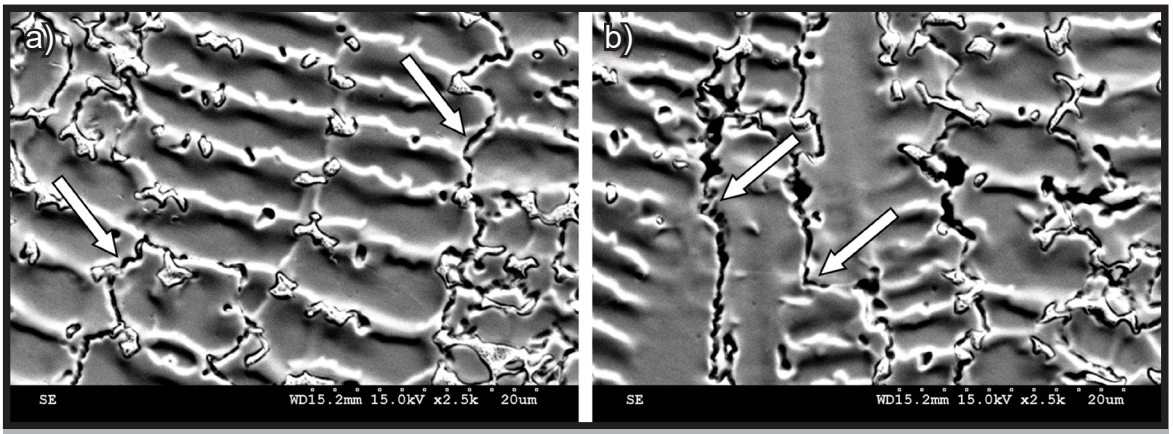

FIG. 3. Microstructure of Co-Cr-Mo alloy obtained by the lost-wax casting method with revealed microcracks in the dendritic microstructure; the images taken for different places on the sample (a, b); SEM, etched state. The arrows show examples of microcracks in the alloy.

The carbide phases are the main source of the alloy's reinforcement [10,32-34]. It is possible to notice the areas (FIG. 3a,b) between the arms of the dendrites, the so-called interdendritic spaces, where carbide phases precipitate. According to the literature data [9,18,32], type M_7C_3 and M_6C, as well as $M_{23}C_6$, carbides can be present in Co-Cr-Mo casting cobalt alloys. The chemical composition analysis performed on micro-areas proved larger segregation of Mo into the interdendritic spaces (FIG. 4c, point 1-3 of the analyses).

The microstructure of the Co-Cr-Mo (EOS CobaltChromeMP1) alloy samples obtained by the direct metal laser sintering technique (DMLS) resembles "fish scales" in the light microscope images (FIG. 2a-d). The traces of the crossing laser beam are clearly visible. The fine-grained microstructure and multi-directionality of the occurring crystallization can be seen (FIG. 2d and FIG. 5b). One can notice that the grain boundaries exhibit geometrical diversity (FIG. 5). Also, the phenomenon of epitaxial dendrite growth is visible - the already present crystal growing through the consecutive melted layers.

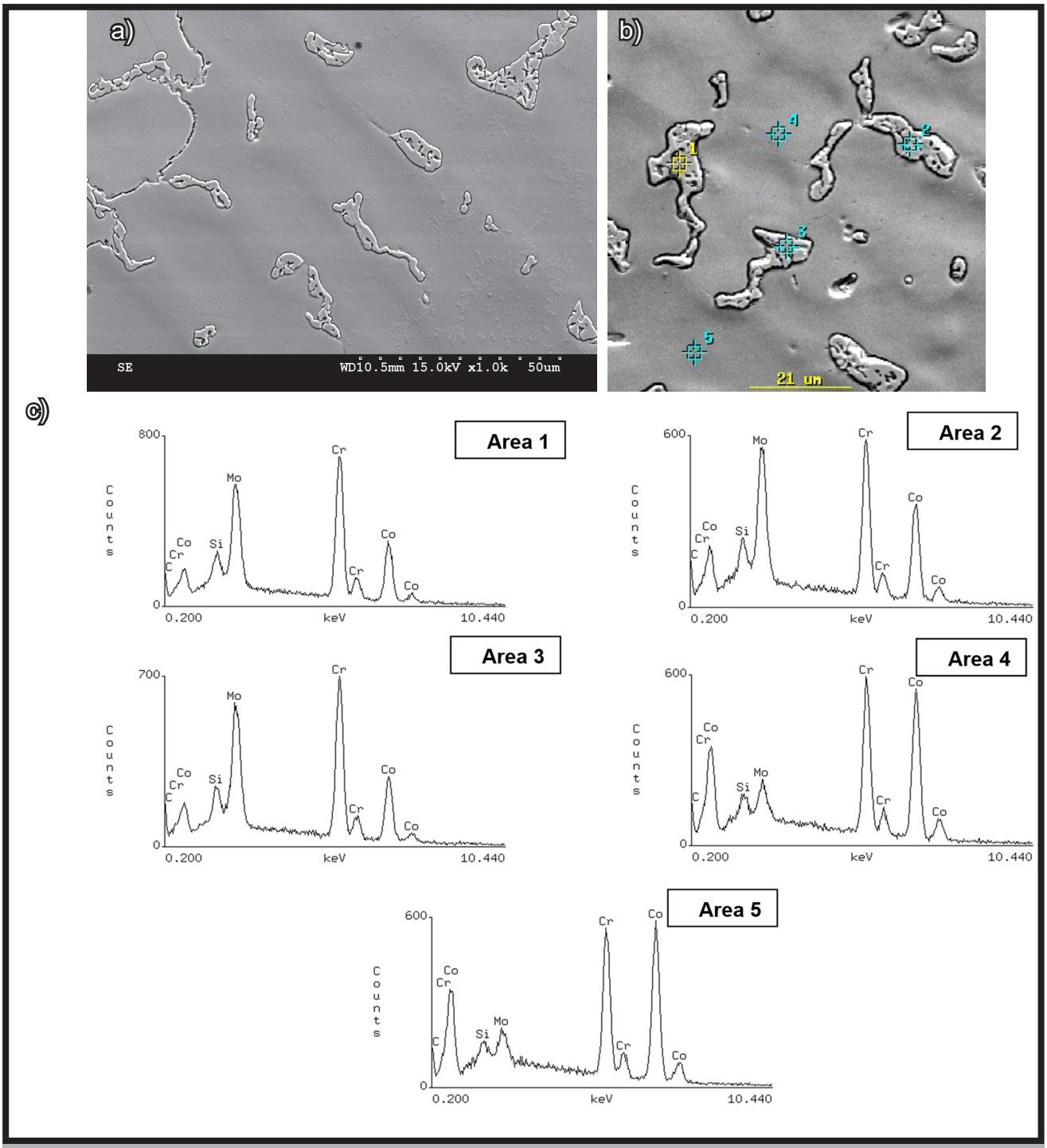

FIG. 4. Microstructure of Co-Cr-Mo alloy obtained by the lost-wax casting, SEM image (a) and X-ray spectra (c) of the places shown in the SEM microphotograph (b).

The dendrite axes formed in the microstructure stay in accordance with the heat removal direction during the crystallization process. The dendrites maintain their directions and run through the grain boundaries (FIG. 5a,b). The main dendrite axes are long and not much branched, which proves that the directional crystallization proceeds at a high rate [35,36]. The Co-Cr-Mo alloy samples obtained by DMLS have a very fine-grained microstructure which provides good mechanical properties of the alloy. Cobalt ensures high hardness, abrasive resistance and bending strength. The presence of chromium and molybdenum provides the alloy with good corrosion resistance.

The results of the EDS chemical composition analysis performed on the microsections of the Co-Cr-Mo (EOS CobaltChrome MP1) alloy obtained by the DMLS method are presented in FIG. 5 in the form of X-ray spectra. The EDS analysis of the sample's micro-areas did not reveal visible differences in the element content in the examined points, which confirms the homogeneity of the examined Co-Cr-Mo alloy, also established in the study [37]. No precipitation of carbide phases was observed for the samples obtained via the DLMS technique. During the DLMS process, the applied Co-Cr-Mo (EOS CobaltChrome MP1) powder was melted down.

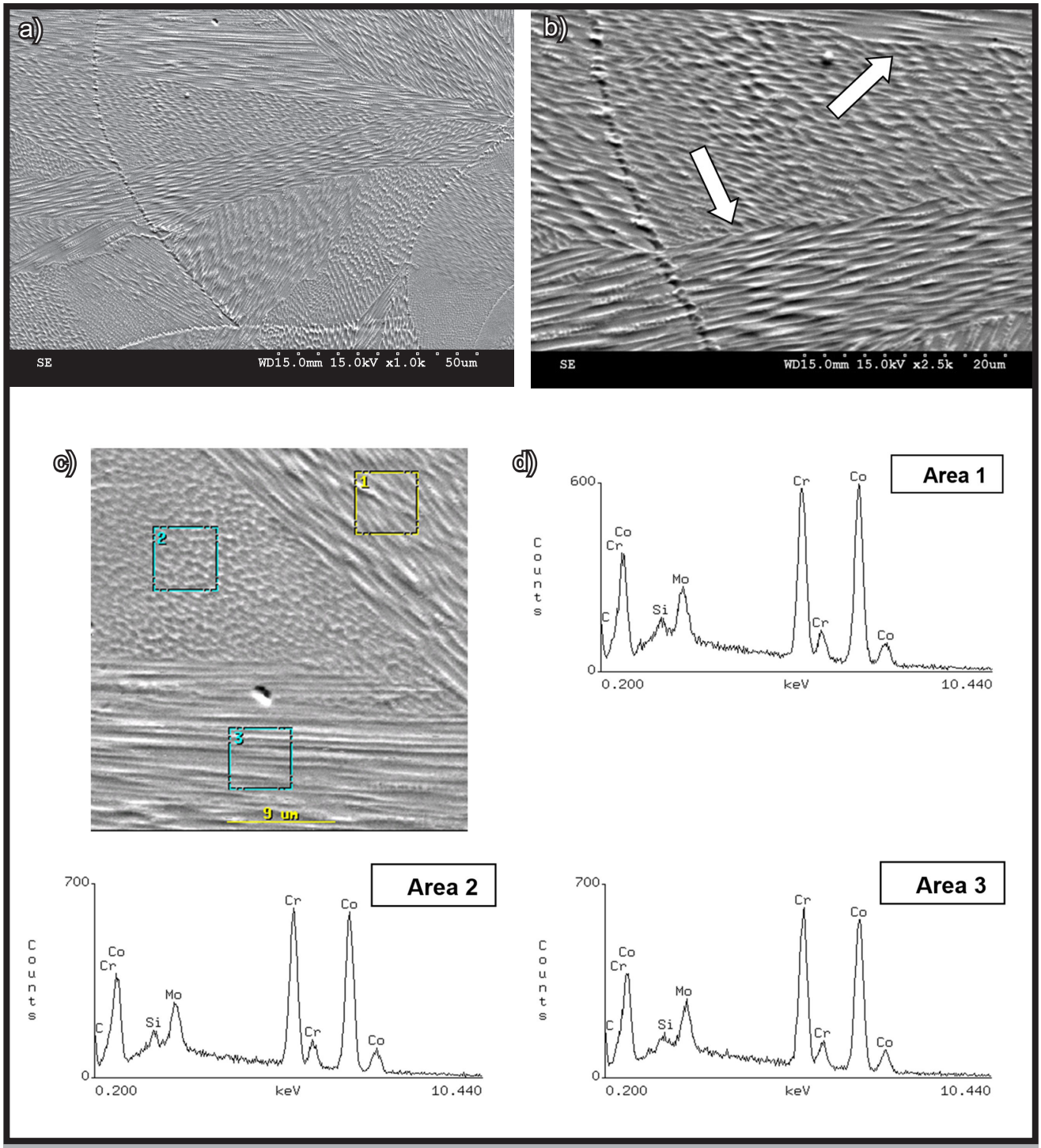

FIG. 5. Microstructure of Co-Cr-Mo alloy obtained by DMLS technique, SEM images (a, b) and X-ray spectra (d) of the places shown in the SEM photomicrograph (c).

TABLE 5. Hardness test results ($\overline{HV10}$) of Co-Cr-Mo alloys obtained by the lost-wax casting method and the DLMS technique.

Co-Cr-Mo samples obtained in the method	$\overline{HV10}$	$S_{(\overline{HV10})}$	δ_g	γ_g [%]
Lost-wax casting method	365	6.5	12.9	3.5
DLMS technique	474	5.4	10.6	2.2

Having in mind the microstructure observation results and the performed investigations [12,14,15] of the possible chemical inhomogeneities of the Co-Cr-Mo alloy obtained by the lost wax method and the finishing treatment (milling, grinding and polishing), the samples of both tested types underwent hardness measurements (HV10) in randomly selected areas. The mean hardness of the cast alloys was calculated, together with the standard deviation, the absolute limiting error (δ_g) and the relative limiting error (γ_g), according to the guidelines presented in the study [38]. A summary of the results is shown in TABLE 5.

The Co-Cr-Mo alloy samples obtained by the lost wax method exhibited the hardness of 366 HV10, whereas the mean hardness of the DMLS alloy equalled 477 HV10. The observed differences in the hardness values result from the fact that no significant differences in hardness in the whole volume are present in the EOS CobaltChrome MP1 alloy as it is chemically homogenous. In turn, the Co-Cr-Mo alloy samples obtained by the lost wax method display the diversified hardness: the lowest values were recorded in their central area, i.e. HV10 at the level of 340-350, while on their edges the values reached 360-370 HV10. This phenomenon is connected with the crystallization and the heat removal, the dendritic microstructure and the lack of chemical homogenization of the cast Co-Cr-Mo alloy.

Conclusions

Based on the performed microscopic observations (light microscopy and scanning electron microscopy, together with an EDS analysis in microsections) and the hardness measurements of the analysed alloys, the following conclusions were drawn:

The examined samples of the alloys Co-Cr-Mo (Wironit LA) and Co-Cr-Mo (EOS CobaltChrome MP1) were characterized by the dendritic microstructure.

The Co-Cr-Mo alloy samples had the matrix (dendrites) consisting of a chromium and molybdenum solution in cobalt, whereas, precipitations of carbide phases were present in the interdendritic areas, resulting from the chemical segregation of Mo and Cr.

The microstructure of the Co-Cr-Mo (EOS CobaltChrome MP1) alloy samples obtained in the DMLS technology exhibited fine grains and multi directionality of the alloy's crystallization. Also, the epitaxial growth of the dendrites was observed.

The samples obtained by the DLMS were characterized by the hardness higher than the cast alloy Co-C-Mo samples. The hardness values were directly affected by the fine-grained and more homogeneous microstructure of the examined DLMS material, as compared to the lost wax cast samples.

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