STRUCTURE OF MULTI – LAYER METAL MATERIALS

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SUMMARY

Multi-metal functionally graded specimens of a CoCr alloy and AISI 316 steel were prepared by MMS – Multi-Material Sintering and 3D-Printing. The processing conditions and powder size varied. The size distribution and shape factor of pores were determined by image analyses. Element distribution and interface thickness were determined by X-ray analysis. Microhardness was measured across the multi-material interface. Optimum processing conditions with regard to porosity and microhardness were identified.
Key words: Sintering, metal powder, biomaterial, interface

1. INTRODUCTION

Recently there has been a shift from rapid prototyping to rapid production of real products which are customized to needs of individual customers. In this respect multi-material functionally graded sintering has been introduced into production of materials. Mechanical, physical and chemical properties change from one side of the product to the other (site specific properties). In particular, this technology is suitable for rapid production of bioimplants. Biomaterials with excellent osteointegration and low Young's modulus on one side of the implant and other materials with perfect mechanical properties such as hardness, abrasion resistance etc. on the other side are promising for various bio-applications. For example, the disadvantage of joint requirements in bioimplants can thus be eliminated and the risk of screw connections or corrosion of weldings avoided.

An ideal material or combination of materials for hard tissue replacements should have the following properties: 'biocompatible' chemical resistance, excellent corrosion resistance, acceptable strength, low Young's modulus and high wear resistance [1], [2]. Another essential property of the implant is its surface structure. Controlled surface roughness and porosity are a key to excellent osteointegration.

The above conditions are fulfilled for certain types of materials. Fundamental materials especially for joint and bone implants are pure Ti, Ti-6Al-4V alloys, Co-28Cr-5Mo alloys and 316L stainless steel. These materials can also be used in metal powder production. Development of biomaterials for powder metallurgy has recently received much attention. New compositions and sintering process parameters are widely investigated in order to produce optimal microstructures [3-8].

The purpose of this work was to verify the internal structure of multi material functionally graded specimens produced from a CoCrMo alloy and 316L stainless steel alloy under various processing conditions. The main aim was to evaluate their mechanical properties and to recommend the best processing-property profile for further research in the field of cytotoxicity.

2. MATERIALS

Two different kinds of materials were used for the production of multi-metal functionally graded specimens (10mm in diameter and 20mm height). The first one was an ASTM F75 Co-Cr-Mo alloy, marked in figures as Co-part. The second material was corrosion resistant AISI 316L steel, marked in figures as Fe- or Steel- part. The Co-Cr alloy formed the bottom of the specimens and the stainless steel the top as shown in Figure 1.

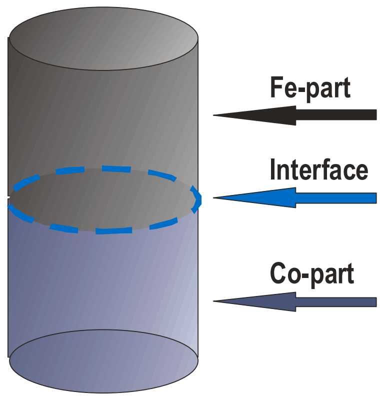

Figure1: Multimaterial sintered sample

The types of materials and processing conditions are shown in detail in Table 1. The chemical composition of both powder materials is given in Table 2.

Specimen code	Upper part/grain size	Sintering temperature [°C]	Process	Atmosphere
452	316L/31µm	1280	MMS	Argon
453	316L/105µm	1280	3DP	Argon
454	316L/31µm	1280	3DP	Argon
458	316L/105µm	1320	MMS	Argon
459	316L/31µm	1320	MMS	Argon
461	316L/31µm	1320	3DP	Argon

Table 1: Processing parameters of investigated specimens

Material	Composition										
	Fe	Mn	P	S	Si	Cr	Ni	Mo	C	N	Co
316L	bal	2,00	0,045	0,030	1,00	17	12	2,5	0,03	-	-
F75	0,16	0,53	-	-	0,95	29	0,12	5,6	0,2	0,18	bal

Table 2: Chemical composition of powder materials

3. EXPERIMENTAL PROCEDURES

The samples were prepared by pouring two different powders on top of each other into a die followed by co-sintering (MMS), and by 3D-Printing of a polymeric binder into the metal powder layer by layer and changing the powder after printing half of the sample also followed by co-sintering (3DP), respectively. One layer of 3D-printed powder has the typical thickness of 150µm. The main difference of the samples

using the two processes is probably related to different particle packing or arrangement in the green compacts.
Specimens were cut lengthwise and the inner surfaces polished using diamond paste. Impurities and diamond paste residues were removed. Pictures of the microstructure were made with a JEOL JSM 5410 scanning electron microscope. Porosity was evaluated from the microstructures at a 50x magnification. The pore size distribution and their shape factor were determined using LUCIA 3D software. Porosity was defined as the ratio of the pore area to the area of the analyzed region, which relation is further denoted as the Pore Area Fraction (PAF). Another factor which was taken into account was the rate of the area of pores, which gives the pore size distribution in the analyzed region. Microhardness tests were performed with LECO M-400-G1 measuring equipment. The chemical composition and element distribution were obtained by WDS X-ray analysis using Link AN 85S and CAMEBAX analytical systems.

4. POROSITY - RESULTS

The porosity of the interface between the cobalt-chromium and the stainless steel parts of the specimens were closely observed. Comparing the interface and the bulk material, only slight discrepancies in porosity (independent of the analyzed place) were observed. The average values of the Pore Area Fraction (PAF) of the Fe-part and Co-part in each specimen were calculated and compared in Figure 2.

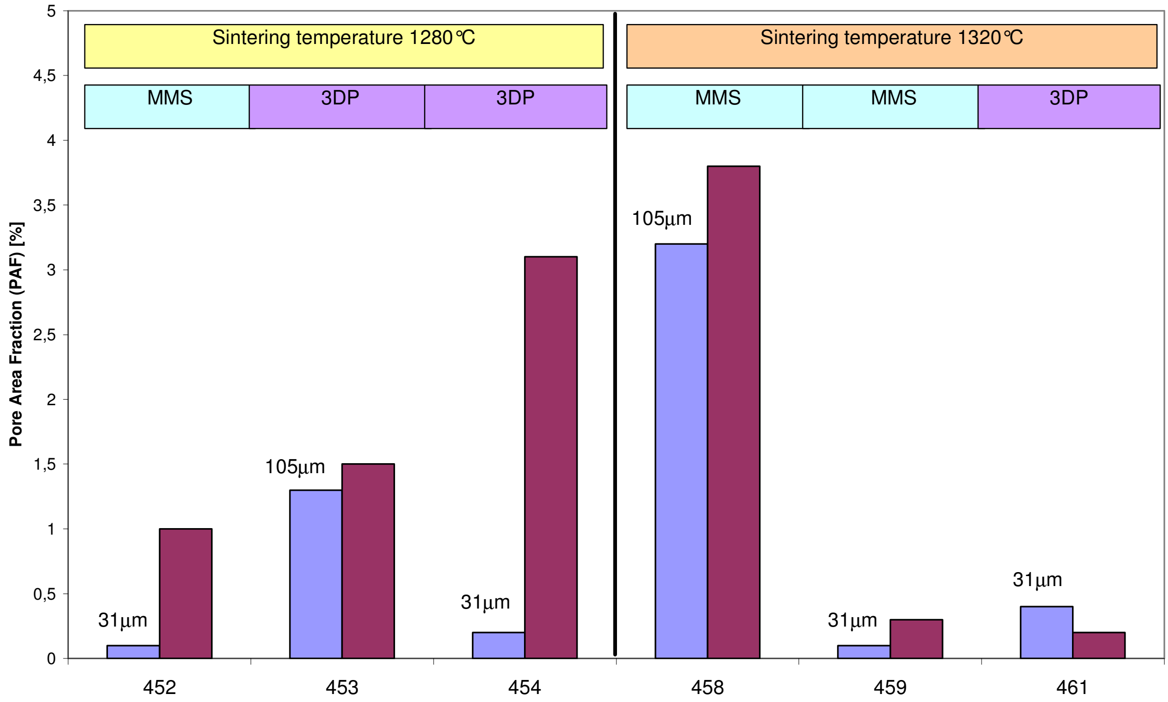

Figure 2: Results of porosity measurement - Pore Area Fraction (PAF)

Figure 2 shows that porosity of the Fe-part decreases when a powder with smaller grain size (31 µm) is used. Lowest porosity can be seen in specimens 452, 459 and 461. A higher sintering temperature (1320°C) improves the porosity of the Co-part from 1% (specimen 452) to 0.3% (specimen 459) or from 3.1 (specimen 454) to 0.2

(specimen 461), while the Fe-part remains almost unaffected. For both materials the MMS method is more effective in improving porosity than the 3DP process. This is apparent when comparing specimen 452 (PAF 0.1% for Fe- and 1.0% for Co part where the MMS method is applied) and specimen 454 (PAF 0.2% for Fe and 3.1% for Co-part where the 3DP process was applied) or specimens 459 and 461. Furthermore, histograms of the pore area (not presented here) show that porosity is predominantly formed by smaller pores, in other words small pores are more frequent rather than large ones. This conclusion promises good mechanical behaviour since no large defects are present in the bulk material.

5. MICROHARDNESS - RESULTS

Measurement of microhardness was highly influenced by the porous structure. Average values of five measured positions in every row for each specimen are summarized in Figure 3.

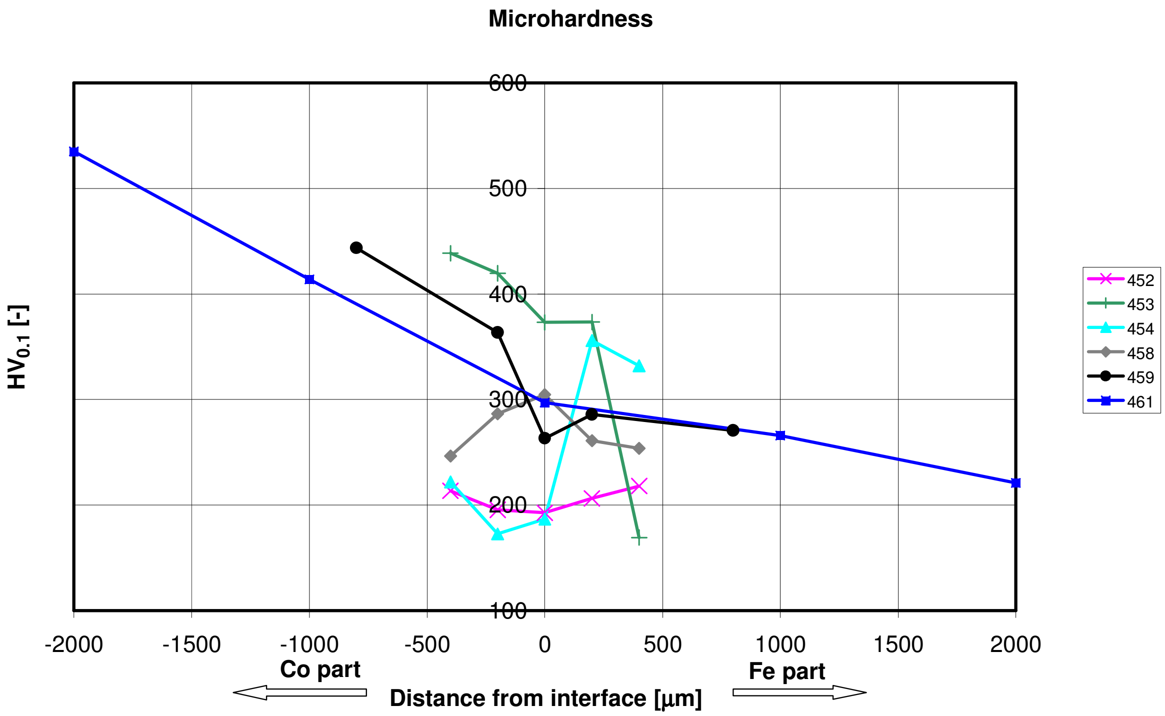

Figure 3: Results of microhardness measurements

The dependances show that hardness generally decreases gradually across the interface from the Co-part to the Fe-part. Microhardness of the interface is highly influenced by the grain size of the stainless steel powder. For example specimens 453 and 454 (or 458 and 459) which were both produced under the same conditions show that material with a smaller grain size "penetrates" into the material with a larger grain size. Therefore e.g. specimen 454 exhibits higher values of microhardness in the steel part and lower values in the cobalt-chromium part. The MMS or 3DP process has no significant effect on the specimen microhardness. Powder grains are not influenced by the manufacturing process. However it is very difficult to determine which is the major processing parameter leading to an improvement of hardness. The most promising hardness profile can be seen in specimens 459 and 461. In both specimens smaller grain size of the steel part was used and the sintering temperature was 1320℃.

5. X-RAY ANALYSIS - RESULTS

The composition of the specimens across the interface and consequently the interface thickness were determined by X-Ray analysis.
The average value of interface thickness for every specimen is given In Table 3.

Specimen number	Powder size of 316L [μm]	Sintering temperature [℃]	Manufacturing Process	Interface thickness [μm]
452	31	1280	MMS	446
453	105	1280	3DP	580
454	31	1280	3DP	580
458	105	1320	MMS	773
459	31	1320	MMS	515
461	31	1320	3DP	3400

Table 3: Results of interface thickness measurement

By comparing results of X-Ray analysis it can be inferred that the 3DP process increases the interface thickness in contrast to MMS namely at a higher temperature (1320℃) . The smaller grain size of stainless steel also extends the interface. The thickness of the interface grows intensively with the sintering temperature and number of sintering cycles. Diffusion affects the compound composition namely at higher temperatures and longer times.

6. CONCLUSIONS

Cylindrical multi-material functional specimens made of an ASTM F75 cobalt-chromium alloy and AISI 316L stainless steel were investigated to determine their porosity, microhardness, composition and interface thickness in relation with their manufacturing conditions.
The following conclusions can be made on the basis of measurements:

- Lowest porosity was observed in specimens 452 and 459 (both produced by multi-material sintering with a smaller grain size of the stainless steel). In general multi-material sintering was more effective in improving porosity compared with 3D-Printing. The sintering temperature also played a significant role. Small pores were more frequent than large ones which could result in better fatigue resistance. Smaller stainless steel powder grain size was also beneficial to porosity.
- The most promising hardness profile was proven in specimens 459 and 461. However, it was difficult to identify the major processing parameter leading to an improvement of hardness.
- Sintering temperatures and the number of sintering cycles were the most important features affecting growth of the interface thickness. Diffusivity of the components significantly affected the interface thickness. Also 3D-Printing supported growth of the interface thickness.

From the above conclusions it can be recommended to use smaller powder grain size in sintering processes of bioimplants. Furthermore, higher temperatures are advantageous to obtain higher densities.
Rapid prototyping appears to be a promising technology for multi-material functionally graded specimens in the design of bioimplants. Mechanical properties are appropriate for bio-applications. Ultimately, further experimental work is highly recommended, especially with respect to fatigue resistance and biocompatibility.

Acknowledgement

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