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Full Length Article

The correlation between substrate and deposited biocompatible layer microstructures on different substrates

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ABSTRACT

The biocompatibility is essential for the so called biomaterials used for medical devices (e.g. artificial implants, implants). It is determined mainly by the surface chemical composition and the environment in which the implant is used. The implant materials used till the date have limited biocompatibility. On the other hand newly developed biocompatible materials are usually more expensive. The biocompatibility of currently used materials can be improved via deposition of thin biocompatible layer on the substrate. This layer may be further improved (or used as interlayer) introducing hydroxyapatite, $BaTiO_3$ or other surface treatments. The microstructure of deposited layers has to be known in such cases as it can influence properties of the surface. Various substrates (pure titanium, Ti6Al4V alloy and Ti-39Nb alloy) were used for biocompatible Ti-39Nb layer sputtering deposition in current paper. The microstructure of substrates has been characterized using electron microscopy techniques. The correlation between substrate and layer microstructures could have been revealed, because both microstructures were studied at the same area of the substrate. Significant differences in grain size have been observed on various substrates. Also the microstructure of deposited layer can be distinguished into two types. These are pure β-Ti phase probably on the grains where the surface diffusivity is high enough (locally observed on Ti-39Nb and pure Ti substrates) and mixture of α″ martensitic and β-Ti phase columns.

1. Introduction

The demand for new biomaterials increased significantly last years. It is due to a fact that many young people need implants after injuries and moreover the lifespan has also increased. Therefore the time for which the implant is in use is also prolonged. Biocompatibility is essential property for materials intended to be used as implants. As the material is in continuous and long term contact with the human body tissues and fluids, it should be fully biocompatible. The term biocompatibility has wide meaning as defined by Williams et al. [1]. We can define biochemical compatibility, biomechanical compatibility etc.. It also depends on intended use (i.e. biodegradable materials, materials for long term use) of the material. Especially the materials for long term use have to contain only fully biocompatible and non-toxic elements [2]. All potentially harmful elements should be avoided (e.g. V, Al, Cu, Fe). One of the ways how to improve the biocompatibility of the currently used materials (e.g. Ti-6Al-4V, stainless steel) is to protect the material from contact with body tissues and fluids. This can be ensured through biocompatible barrier (layer) formed on the surface of implant. This can, however, improve only the biochemical compatibility but, the biomechanical compatibility remains nearly the same. The biomechanical compatibility means that the material has mechanical properties similar to those of the human bone. Especially low Young's modulus is important as in this case. If the modulus of the implant is significantly higher than the bone modulus the so called stress shielding effect could emerge [3,4].

The properties of the deposited layer are influenced by many parameters. Among those the used materials (both substrate and layer), deposition method and deposition parameters should be considered to influence the resulting microstructure [5–7]. Also the substrate itself may influence the microstructure (and possibly resulting properties) of the layer. Adhesion between the substrate and the layer is very important. All those properties determine whether the layer may significantly improve the implant biocompatibility. In current work we investigated, whether there is any relation between the substrate and the layer microstructures.

2. Experimental methods

Three different substrates were prepared for thin layer deposition. Ti-6Al-4V alloy (in this work named as "Grade 5"), pure titanium grade 2 "Grade 2" and Ti-39Nb alloy "TiNb" (all compositions in this work are

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in wt.% unless otherwise noted) were used. Commercially available materials were in most cases used as substrates – typically round bars of approximately Ø10mm. The TiNb substrate was prepared by vacuum arc melting with non-consumable tungsten electrode in water cooled copper crucible. The alloy was remelted six times to ensure good chemical homogeneity. As-cast alloy was homogenized in a vacuum furnace 1000 °C/6 h. Homogenized ingot was subsequently hot forged into a cylindrical rod with a section reduction of about 40%. Hot forged rod was solution treated 850 °C/0.5 h and water quenched. The surfaces of all substrates were grinded up to #4000 with SiC papers and polished with Struers OP-S emulsion. Electropolishing using 1000 ml C_2H_5OH + 50 ml $HClO_4$ + 15 ml HNO_3 electrolyte working at room temperature has been used as a final surface treatment.

Thin biocompatible layer of Ti-39Nb beta titanium alloy with intended thickness of about 2 μm has been deposited on all substrates. The TiNb layer was prepared by cathodic sputtering in a Flexicoat 850 unit (Hauser, Netherland). The deposition time was 2.5 hr at the substrate temperature of 350 °C. Samples were positioned on rotating tool and rotated 2 rpm. The working argon pressure was 0.2 Pa and the substrate bias was 70 V. Deposited layer has been studied in as-deposited state with no post treatments. Additional slight polishing with Struers OP-S emulsion has been performed in some cases (mentioned in the following text).

The microstructure has been studied by light microscopy (LM) using ZEISS Neophot 32 microscope. Scanning electron microscopy (SEM) observations have been carried out on JEOL JSM 7600F equipped with electron back scatter diffraction (EBSD) detector (Nordly's – Oxford instruments, UK). Transmission electron microscope (TEM) JEM2000 EX (JEOL, Japan) working at 160 kV was used for detailed observations on cross sections of the deposited TiNb layer. Specimens for TEM observations were prepared using the JEOL Ion Slicer device.

3. Results and discussion

The microstructure of substrates can be seen in Fig. 1 where the maps of inverse pole figures (IPF) are shown. It is evident that the substrates have significantly different grain size as can be also seen in Table 1 where the their approximate grain sizes are presented.

The microstructure "Grade 2" and "TiNb" specimens consists of relatively equiaxed grains. It was confirmed by EBSD analysis that those phases are α-Ti (hcp) and β-Ti (bcc) for "Grade 2" and "TiNb" substrates, respectively. The "TiNb" substrate has very coarse grains in comparison with all other substrates (Table 1). The "grade 5" substrate consists mainly of equiaxed α-Ti grains with low β-Ti phase fraction. The grain size of α-Ti grains is comparable to that of "Grade 2". No distinct preferable orientation (texture) has been revealed by EBSD analysis in any of studied specimens. It should be pointed out that the results of texture analysis is possibly not representative in "TiNb" substrate due to its large grains and therefore small number of analysed grains.

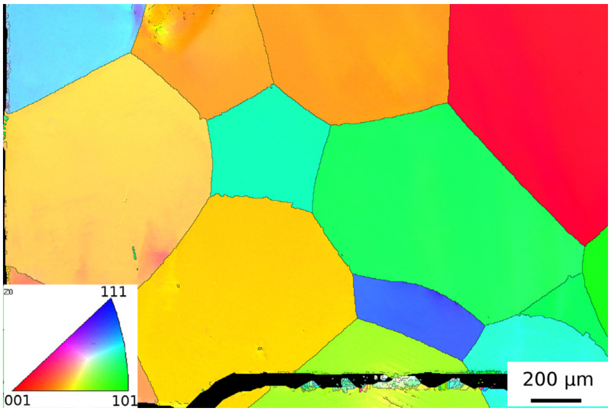

Fig. 1a. Inverse pole figure (IPF) map of "TiNb" substrate.

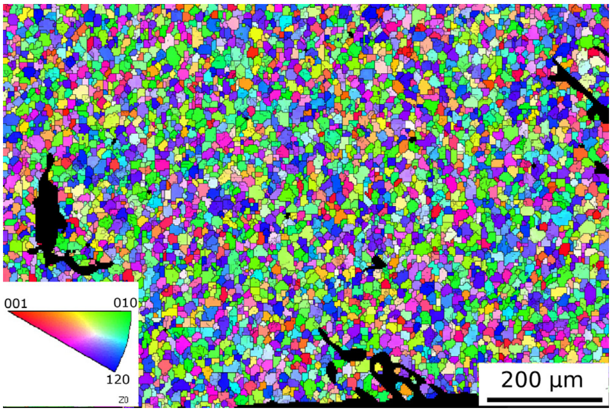

Fig. 1b. Inverse pole figure (IPF) map of "Grade 2" substrate.

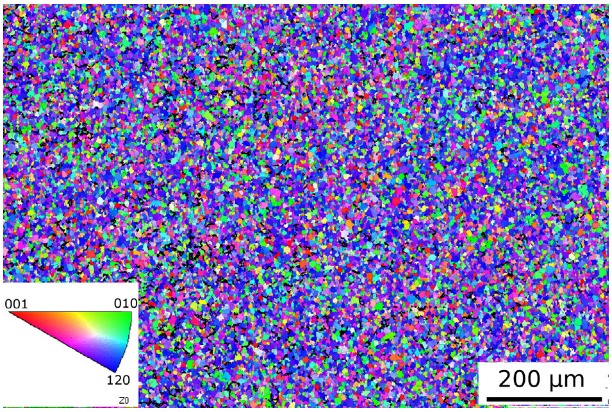

Fig. 1c. Inverse pole figure (IPF) map of "Grade 5" substrate.

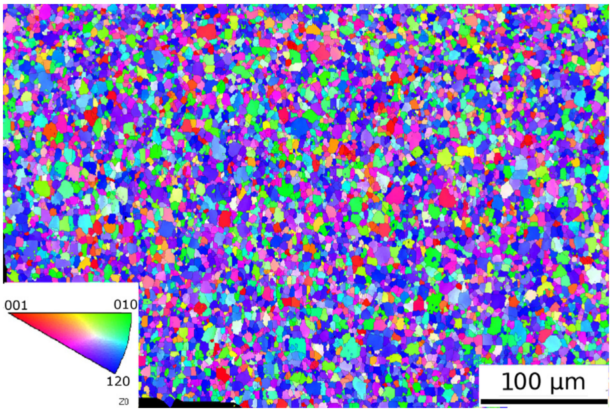

Fig. 1d. Inverse pole figure (IPF) map of "Grade 5" substrate – detail.

Table 1
Grain size of various substrates.

Substrate	Grain size [μm]	remark
"Grade 2"	10	–
"Grade 5"	5	β-phase needles not measured
"TiNb"	600	–

The EBSD maps of as-deposited layers obtained from the same area as in Fig. 1 are presented in Fig. 2. After TiNb layer deposition the Kikuchi bands were visible only locally on films deposited on "Grade 5"