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The role of strain rate during deposition of CAP on Ti6Al4V by superplastic deformation-like method using high-temperature compression test machine

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Abstract

This paper describes an implementation of superplastic deformation method for the deposition of carbonated-apatite (CAP) on the well-know titanium alloy, Ti6Al4V. This deposition process was carried out using high-temperature compression test machine, at temperature of 775 °C, different strain rates, and conducted along the elastic region of the sample. Before the process, titanium substrate was cryogenically treated in order to approach superplastic characteristic during the process. After the process, thin film of CAP was created on the substrate with the thickness from 0.71 μm to 1.42 μm. The resulted film has a high density of CAP that covered completely the surface of the substrate. From the stress–strain relation chart, it can be observed that as the strain rate decreases, the area under stress–strain chart also decreases. This condition influences the density of CAP layer on the substrate that as this area decreases, the density of CAP layer also decreases as also confirmed by X-ray diffraction characterization. In addition, since the resulting layer of CAP is in the form of thin film, this layer did not alter the hardness of the substrate as measured by Vickers hardness test method. On the other hand, the resulting films also show a good bonding strength properties as the layer remain exist after friction test against polishing clothes for 1 h.

Keywords: Deposition process; Carbonated-apatite; Titanium alloy; Superplastic deformation; Strain rate

1. Introduction

Ti6Al4V has been widely accepted as implant material in the medical area application [1]. Its resistance against oxidation and corrosion make this material is a good choice for this application [2]. Furthermore, the ability of this material to be deformed superplastically [3] makes this material as the preferable material to substitute complex shape hard tissue. Material in the superplastic condition is easier to be deformed since the flow stress is relatively low [4]. Thereby, a complex shape of product is possible to be produced.

However, Ti6Al4V alone does not fully satisfy the biocompatibility requirement for material to be used as implant product. Therefore, in most cases, ceramic bio-apatite such as hydroxyapatite (HAP) or carbonated-apatite (CAP), normally is coated on this alloy for the above purpose. Beside improvement in the biocompatibility, coating of the implant product also improves the mechanical properties of the implant. Having ceramic bio-apatite on the titanium substrate will produce a good toughness implant product by having a hard surface and ductile substrate as well.

Numerous methods have been employed to produce bio-apatite coated on titanium substrate [5–8]. Among them, plasma spray has been the most popular method for the coating process of bio-apatite on titanium substrate [9]. However, the bonding strength of bio-apatite on the substrate remains a challenging task to be solved at the current progress. Therefore, the present work tries to solve this problem by conducting deposition of CAP using continuous pressing method at elevated temperature which can be considered as superplastic deformation-like method. By this method, it is expected that beside diffusion process occur from high-temperature condition of the process, the additional pressing work can give additional energy that force the bio-apatite to move inside the substrate and in turn enhance a good bonding properties of bio-apatite on the substrate.

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2. Experimental work

Ti6Al4V with the size of 1 cm × 1 cm × 2 cm was selected as the substrate material, whereas CAP powder was selected as the deposited material. Before the process, substrate was cryogenically treated by liquid nitrogen spraying after heating at 950 °C. The objective of this treatment is to obtain suitable superplastic condition of the substrate during the deposition process. On the other hand, CAP powder source which is supplied by AMREC SIRIM Berhad Malaysia, was ground using grinding ball for 8 h and sieved to the sieving mesh of 53 μm. The CAP powder that is used for the deposition process is the powder that passed the sieving mesh. Therefore, the powder size is below 53 μm before the process.

Deposition process was conducted using compression test machine (Instron) that is equipped with high-temperature furnace and controlled gas atmosphere. Argon gas was used as the controlled gas atmosphere while the process was conducted at temperature of 775 °C. Strain rate was varied at $10^{-4\circ}\,s^{-1}$, $2\times10^{-4\circ}\,s^{-1}$, $4\times10^{-4\circ}\,s^{-1}$, $8\times10^{-4\circ}\,s^{-1}$ and $16\times10^{-4\circ}\,s^{-1}$. Since the objective of the experimental works is deposition process and not a deformation process, it should be avoided plastic deformation to take place during process. Therefore, experimental work was conducted at the elastic region of the compression process of the sample. This condition was done by stopping the compression process at around the yield point of the process. This method of using Instron machine for the deposition process also gives an advantage that a direct observation in term of stress–strain behavior during the process can be evaluated. Fig. 1 shows the schematic figure of the compression system used in this research.

Several characterization processes were conducted after the deposition process. In order to investigate microstructure of the CAP layer, optical and scanning electron microscope observation were conducted. X-ray diffraction analysis was also conducted in order to study crystal structure of the resulting CAP layer. Vickers hardness test was also conducted in order to evaluate the hardness of sample that is covered and uncovered with CAP layer after deposition process. In addition, in order to test the durability or bonding strength of the resulting CAP layer, friction test was also conducted using polishing machine with the speed of rotation at 250 rpm. This test was conducted against polishing clothes under water spraying condition.

3. Results and discussions

3.1. X-ray diffraction analysis and microstructure characterization

Fig. 2 shows electron micrograph of the cross-section of the CAP layer on titanium substrate. The white layer is CAP thin film with the range of thickness from 0.71 μm up to 1.42 μm. For the high strain rate sample (Fig. 3(b and c)), high density of CAP films can be obtained and CAP layer cover completely the microstructure of titanium alloy substrate. For the lower strain rate sample as is shown in Fig. 3(d–f), low density of CAP film is resulted. As can be seen from these figure that a shallow layer

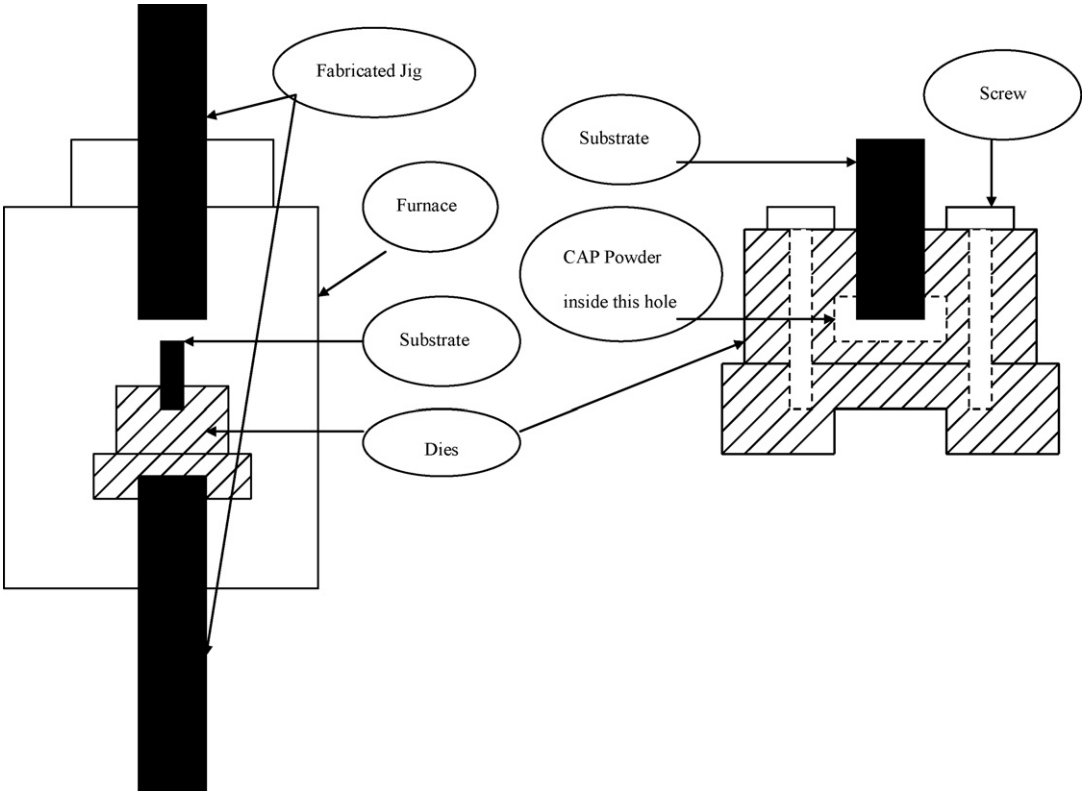

Fig. 1. Schematic drawing of deposition system, the left figure is the system including furnace, and the right figure is the highlight figure of dies, substrate and position of CAP powder.