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Fabrication of a Ti-Al_2O_3 functionally gradient tooth root by dry jet spraying of ultrafine particles

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A Ti-Al_2O_3 functionally gradient tooth root is fabricated by dry jet spraying of ultrafine particles onto the surface of a cylindrical Ti rod. Ti/Al_2O_3 ratio decreases gradually in the outward radial direction from 1 to 0. The green composite is sintered in a temperature gradient condition, where Ti-rich side is sintered at about 1500 K and Al_2O_3-rich side at about 1800 K. No contamination with poisonous heavy metals occurs during these processes. The adhesion strength between the substrate and the spray-coated layer is about 60 MPa. The composite has compressive strength of 200 MPa and is durable against fatigue test of 10^7 stress cycles at 10 N. These mechanical properties are considered to be suitable for dental use.

1. INTRODUCTION

Ultra-fine particles (UFPs) of submicron sizes are known to exhibit various interesting physical, chemical and mechanical properties [1,2]. Besides individual UFPs themselves, bulk materials composed of UFPs are also expected to have peculiar properties such as superplasticity and ultrafine porosity. The applications of UFPs have been tried in several fields such as catalysis, gas sensing, electronics, magnetism. Only a few, however, have been successful from the industrial points of view.

In this work a trial to fabricate a functionally gradient material (FGM) with the use of UFPs is undertaken. An artificial tooth root is chosen as an example of FGMs. It consists of a cylindrical core rod of Ti and a surface coated FGM layer, where the composition ratio, r = Ti/Al_2O_3, decreases gradually in the outward radial direction from 1 to 0. The mechanical strength of the tooth root is sustained by the Ti rod, while the Al_2O_3 surface serves as a biochemically stable (bioinert) layer. If much higher biocompatibility is required, the Al_2O_3 surface is further coated with hydroxyapatite (HAP) [3-6].

2. FABRICATION PROCEDURES

A schematic view of the whole system for the synthesis of Ti-Al_2O_3 mixed UFPs and their deposition by dry jet spraying is shown in Fig. 1. It consists of two raw powder feeders, a plasma chamber, a gas cooler, a deposition chamber and an exhaust pump.

Radio-frequency (RF) plasma is chosen among several dry or physical methods for the synthesis of UFPs because of its contamination-free reaction field and high reaction efficiency [7]. The plasma power is about 40 kW (4MHz) and the flow rates of plasma gasses are 85 l/min for Ar and 15 l/min for H_2.

Raw material powders of Ti (average diameter = 27 μm) and Al_2O_3 (10 μm) are fed into the plasma flame with two mutually independent dispersion feeders, where Ar gas is used as carrier. The raw powders are mixed before they arrive at the feeding nozzle which is located on the top of the plasma chamber. By adjusting the feeding rates of the two feeders the ratio r of the raw powders can be adjusted at any value between 0 and 1. The total feeding rate is kept at 2 g/min.

The produced UFPs are homogeneously mixed in an aerosol form, cooled in the cooler and fed into the deposition chamber. There the UFPs are dry-jet sprayed [8] onto a Ti substrate to form an FGM layer. The cross section of the spraying nozzle is rectangular (2 mm x 20 mm) and the speed of spraying gas is about 200 m/sec estimated at the exit of the nozzle. A newly developed L-shaped nozzle is used to increase the spraying efficiency [9].

The Ti substrate rod of 3 mm in diameter and 40 mm in length is made by cold isostatic pressing (CIP) at about 200 MPa. It is located in front of the nozzle, the distance between them being 15 mm. In order to make the spraying homogeneous the substrate rod is rotated at 30 rpm and simultaneously moved to and fro along the rod length direction with the amplitude of 30 mm and at the frequency of about 0.05 Hz. The total spraying time is 30 min, during which the ratio r of the UFPs changes gradually from 1 to 0. The total thickness of the deposited layer is about 1 mm, which shrinks to about 0.5 mm after sintering.

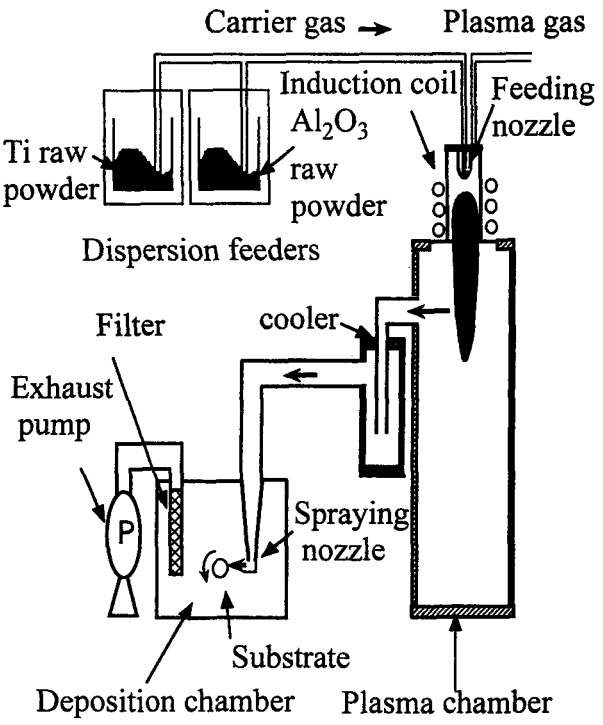

Figure 1. Schematic view of the whole system.

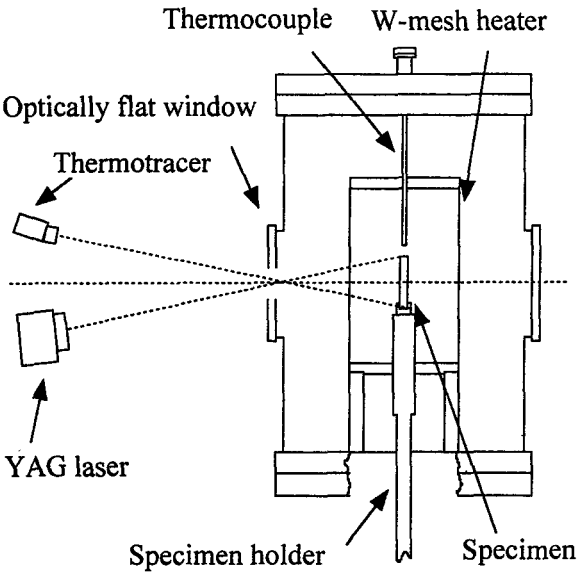

Figure 2. Schematic view of the sintering furnace.

The deposited rod specimen is sintered in a vacuum (10^{-3} Pa) furnace shown in Fig. 2. A W-mesh heater surrounds the specimen to heat the whole part of it at about 1500 K. YAG laser beam is irradiated onto the outermost Al_2O_3 surface of the specimen to heat the Al_2O_3-rich surface at about 1800 K. The temperature difference of about 300 K is attained between the outer and the inner part of the specimen in this way. The specimen rod is rotated at about 10 rpm and the laser is scanned along the rod axis at 2000 mm/sec to make the irradiation homogeneous.

3. RESULTS OF CHARACTERIZATION AND DISCUSION

Since the results of characterization of the UFPs have already been reported [10,11], the details are not given here. An example of scanning electron microscopic (SEM) observation of the cross section perpendicular to the FGM rod axis is shown in Fig. 3. A few cracks observed perpendicular to the layer are due to the difference in the amounts of thermal shrinkage during sintering between the substrate and the FGM layer. Their existence, however, is considered to be favorable for a tooth root, because an osteoblast is known to enter into them to increase the adhesion strength.

The results of electron probe micro analysis (EPMA) of the area enclosed with a rectangle are shown in Fig. 4. Red lines indicate the strength of Ti and Al signals taken from the area between two white lines. It is clearly seen that the ratio r decreases gradually in the outward radial direction from 1 to 0.

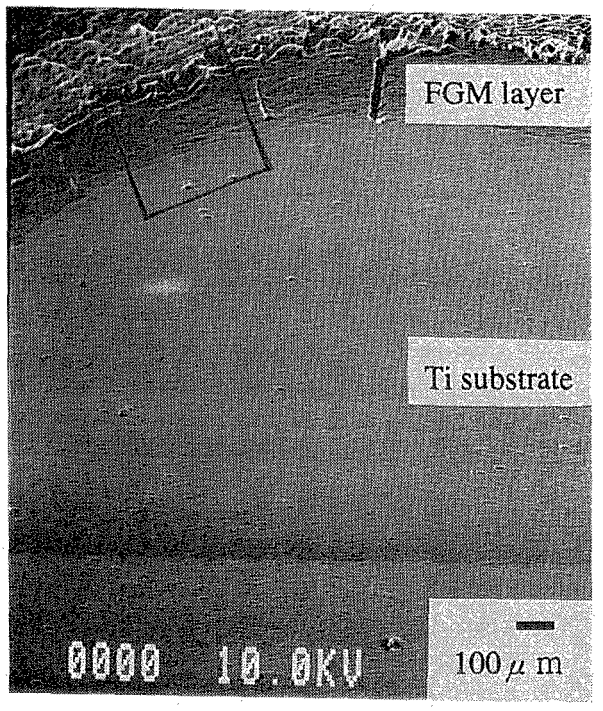

Figure 3. SEM photo of the cross section of the composite.

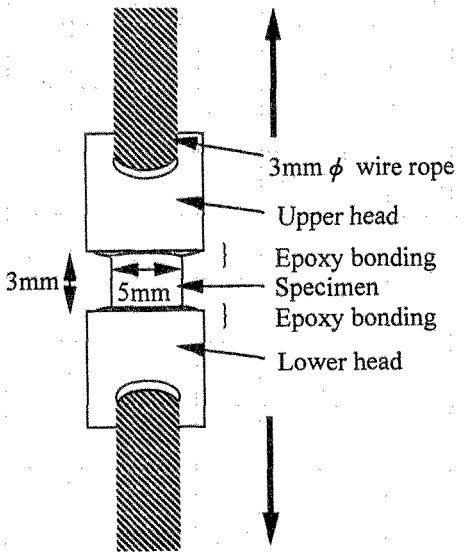

Figure 5. Testing method of adhesion strength.

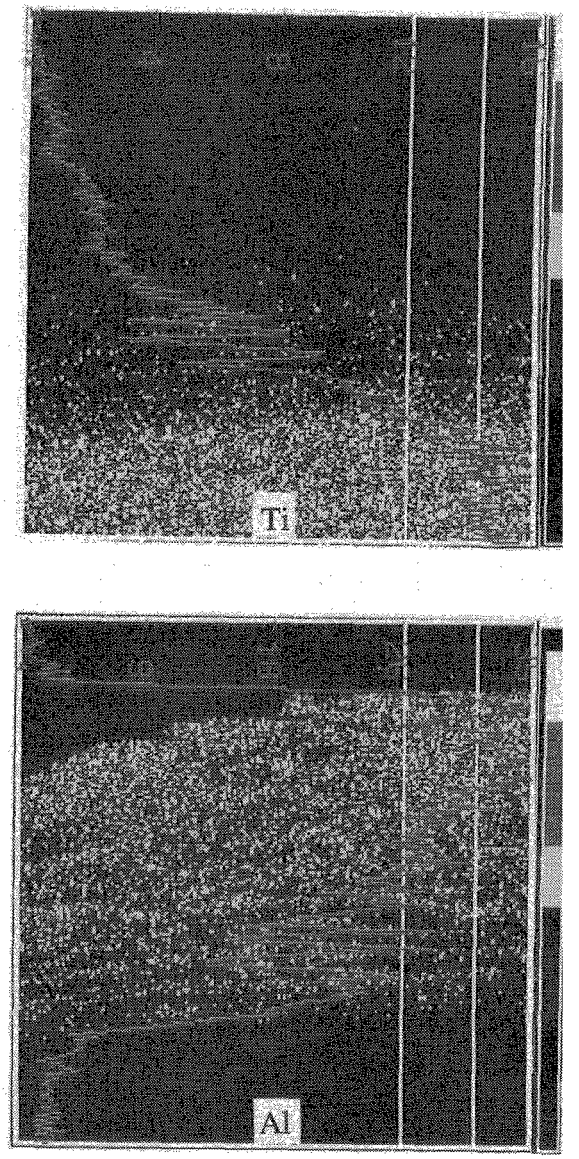

Figure 4. Results of EPMA of the enlarged area of Figure 3.

The adhesion strength between the Ti substrate and the FGM layer is measured by tensile testing as shown in Fig. 5. The specimen is attached to the cross-heads with an epoxy adhesive. During the tensile testing the adhesive breaks at stress levels between about 40 and 60 MPa, which depends on the adhesion conditions. The boundary region between the FGM and the substrate, however, stays undestroyed. This indicates that the adhesion strength between the FGM and the substrate is expected to be higher than 60 MPa.

The compression test in the rod axis direction confirms the compressive strength of more than 200 MPa. The fatigue test is also made by giving compressive stress varying cyclically between 1 N and 10 N at 30 Hz. The composite is found to be durable against 10^7 stress cycles. Analysis by induction coupled plasma spectroscopy (ICP) shows that no contamination with heavy metals poisonous to a human body occurs during the whole set of fabrication processes.

From the results of characterization given above the FGM composite produced here is considered to be highly suitable for dental use.

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