# Surface modification of orthodontic stainless steel with bioactive glass

—Obtaining aesthetic surfaceand enamel remineralization ability—

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【INTRODUCTION】

Many orthodontic materials are formed from metals, which typically have superior mechanical properties compared with other materials, but have less aesthetic nature. In addition, orthodontic fixed appliances creates stagnation areas for plaque, rendering tooth cleaning more difficult, and limits naturally occurring self-cleaning mechanisms. Therefore, the enamel surface around bonded brackets is highly susceptible to demineralization. To cope with these problems, novel method of surface modification for orthodontic metallic materials needs to be developed. Recently, the application of bioactive ceramics to medical treatment has been developing. Especially, bioactive glass(BAG) is known to have excellent bio-affinity and ability of induction into mineralization, and it has been drawing attention for therapeutic application such as artificial bone, dental prosthesis, implant and dental abrasive. Electrophoretic deposition(EPD) is a process that colloidal particles including materials such as metals, polymers, and ceramics, suspended in a liquid medium migrate under the influence of an electric field (electrophoresis) and are deposited onto an electrode. EPD has been considered as a noteworthy method because of its high cost-effectiveness.

The purpose of this study is to investigate whether orthodontic metallic material surface, modified with bioactive glass using electrophoretic deposition has superior mechanical properties and aestheticity or not, and to investigate whether BAG coated layer has important biological properties, such as enamel remineralization ability without cytotoxicity.

【MATERIALS AND METHODS】

**Sample Preparation**

The BAG with composition of $SiO_2$ 46.15 mol%, $Na_2O$ 24.30 mol%, CaO

26.95 mol% and $P_2O_5$ 2.60 mol% was made by melting method at the 1550℃. The BAG was ground in a ball mill to obtain a particle size D50=1.98μm.

The EPD using BAG suspension on stainless steel disk (14.0 mm in diameter with 2.0 mm thickness) and wire having 0.43 mm × 0.64 mm-in cross sections was carried out. The deposition was made at constant voltage of 5 V, 10 V, and 15 V, alternating current(AC) or direct current(DC) on 6 occasions in total.

**Analysis of BAG particles**

Composition was assayed by X-ray fluorescence analysis(XRF). The particles were analyzed by X-ray diffraction(XRD) to clarify their crystal structures. Surface states of BAG particles suspended in aqueous alkaline solutions, heated up to 1000℃ were determined by a potentiometric titration method. Size and shape of BAG particle were observed by Scanning Electron Microscopy (SEM).

**Analysis of BAG layer**

Surfaces and cross-sections of the specimens were observed with SEM and analysis with Energy Dispersive Spectroscopy (EDS) was carried out for checking the distribution of elements such as Si,Ca,Na,P,Fe,Ni,Cr. The surfaces of wire specimens were observed by 3D Laser Scanning Microscopes and the surface roughness was calculated. The colors of samples were measured using a color measurement device.

**Estimation of Caries Preventive Possibility**

Quantity of ion elution in artificial saliva were determined by inductively coupled plasma atomic emission spectroscopy(ICP-AES).

Disk specimens were immersed in acetic acid solution (pH4.5) for 7 days. Time-dependent pH change in the solution was measured using a pH meter during the immersion for acid buffering capacity test.

Human enamel blocks were etched with phosphoric acid gel and immersed in artificial saliva with the specimen disks. Nanoindentation testing of the enamel surfaces was conducted before and after etching, and during the immersion for checking on the extent of enamel remineralization. After immerse period, the enamel surfaces were observed by SEM.

**Cytotoxicity Assay**

Disk specimens were immersed in the culture solution (MEMα+5%CS) containing L929 cells for 24 hours and then the cell proliferation was estimated using Cell Counting Kit-8 for cytotoxicity assay.

**Mechanical Properties of Wires**

Three-point bending tests, friction test and nanoindentation test were performed for investigating the mechanical properties of wires. Color analysis was performed for evaluation of its esthetics.

**Statistical Analysis**

All data were compared using one-way analysis of variance (ANOVA) and the Tukey test, with $P \leqq 0.05$ for statistical significance.

【RESULTS】

**Analysis of BAG particles**

１）XRF

XRF analysis showed coincidence with theoretical composition.

２）X-Ray Diffraction

BAG particles showed amorphous structure.

３） Potentiometric titration method

The isoelectric point couldn't be determined due to slow declining of pH.

４） Examination with SEM

BAG showed round amorphous particles in size of 2 μm to 6 μm.

**Analysis of BAG Layer**

１） Surface observation and compositional analysis with SEM and EDS

Compact Homogeneous layer of BAG was observed at the specimens made at 15 V(DC or AC). And the thickness was 3 to 5 μm.

２） Evaluation of surface roughness

All BG-coated wires showed rougher surface than controls.

３） XRD

The peak signal from amorphous structure was observed in BAG layer.

４） Color analysis

The color of all BAG-coated samples showed most naturally whitish at AC15 V.

**Caries preventive possibility**

１） Acid Buffering Capacity Test

Time elapsed, Acetic acid solution with BAG-coated disks caused increase of the pH time dependently.

２） Enamel Remineralization Behavior Using Nanoindentation Test

The mechanical properties of the etched enamel specimens, immersed with BAG-coated disks recovered significantly, compared to those with the plain disks.

３）Measurement of eluted ions by ICP

Concentration of all ions were significantly higher in the treated samples than in control.

**Cytotoxicity Assay**

There was no significant differences the cell activity between the specimen and the control.

**Mechanical Properties of Wires**

１）Three-point Bending Test

It showed the significantly lower elastic module in all of BAG-coated wires compared with that in the control.

２）Friction Test

The wires BAG-coated at 15V had greater static frictional force compared to the controls. Significant differences was not observed within the BAG coated wires.

３）Nanoindentation Test

In both of hardness and elastic modules, all of BAG-coated wires showed a significantly lower value than the control. The wires with BAG-coated at 15V showed a significantly higher values compared with the wires BAG-coated at 10V.

【DISCUSSION】

Relatively higher voltage condition (10 V, 15 V of DC or AC) was needed to create suitable BAG layers, and for the metallic color of stainless steel covered with more naturally whitish BAG layer. From the results analysed by XRD, amorphous structures and bioactivities were maintained after the treatment with EPD. The BAG layer seemed to have acid-buffering and to enhance remineralization enhancing abilities. The BAG coated wire seemed less exfoliative and more solid for daily use and seemed to have no cytotoxic effect. The surface modification by EDP with BAG improved the esthetic nature of an orthodontic metallic material. BAG coating also prevented demineralization and enhanced remineralization of enamel around brackets during orthodontic treatment.

【CONCLUSION】

This study showed the possibility for developing aesthetic orthodontic metallic materials, maintaining excellent mechanical properties and preventing effect of enamels around brackets against demineralization with BAG by EPD.