

# Nanocoating of Montmorillonite/Mg- $\beta$ -Tricalcium Phosphate on Orthodontic Titanium Miniscrews

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**Abstract**-Mini-implants (1-2 mm in diameter) are miniscrews that are placed in the jaw bones to help Orthodontists move teeth in a controlled manner. The reasons behind their increasing popularity over the past decade is that mini-implants are easily placed and removed under local anaesthetic and can withstand up to 300g force. Unfortunately, their high rate of failure (10-30%) poses a problem to clinicians and their patients alike. Advances in the field of nanotechnology presented a wide range of solutions to biological problems. We developed a nanocoating of nanoclay reinforced magnesium substituted  $\beta$ -TCP on titanium surface to enhance the stability of orthodontic miniscrews.

## I. INTRODUCTION

Temporary anchorage devices (TADs) are small (1-2 mm in diameter) miniscrews that are bone-borne and provide absolute anchorage in orthodontics. They can withstand forces up to 300 grams and therefore are suitable for most orthodontic applications. Even though TADs are gaining importance as an alternative source of intra-oral anchorage, their failure rates of 10–30% as described in the literature poses a clinical problem [1]. The failure of TADs is related to the primary stability and partial osseointegration of titanium miniscrews. Primary stability is the stability of the implant immediately after insertion. Bone quality, implant design, and insertion modalities are the fundamental factors affecting primary stability [2]. Osseointegration, the direct structural and functional connection between living bone and the surface of a load-bearing implant, could affect the long term stability of miniscrews. Recent studies showed that miniscrews used for temporary anchorage in orthodontics undergo partial osseointegration [3]. Thus, we hypothesize that primary stability and partial osseointegration of TADs could be achieved by the synergistic effects from nanoclay reinforced  $\beta$ -tricalcium phosphate (TCP) nanocoating on titanium miniscrews.

Nanoclay exhibits excellent mucoadhesive ability [4] and exceptionally high mechanical properties (Young's modulus calculated at ca. 250-260 GPa [5]) that could positively enhance the primary stability of miniscrews. The nanocoating is expected to enhance the degree of osseointegration by imparting nanostructures to TADs.

Magnesium substituted  $\beta$ -TCP (Mg- $\beta$ -TCP), a biodegradable calcium phosphate, could dissolve in physiological media and be replaced by bone during implantation which will increase the degree of partial osseointegration of miniscrews. Meanwhile the nanostructures of nanocoating could prolong the miniscrews' retention time by adding nanoporosity as well as nanoroughness to the surface of the treated TADs. Nanoporosity and roughness have been shown to improve osseointegration through the development of enhanced coordinated functions of osteoclasts and osteoblasts [6]. A nanoemulsion coating method will be applied to impart nanostructures on titanium miniscrews surface.

## II. MATERIALS AND METHODS

*Preparing of nanoclay suspension.* The nanoclay used is Na<sup>+</sup>-montmorillonite ("Cloisite Na<sup>+</sup>") powder (Southern Clay Products, TX, USA). The nanoclay suspension was prepared by dissolving clay powder in DI water under vigorous stirring for 1 week prior to use.

*Preparing of oil and water phases for making the nanoemulsion.* An acetone solution of calcium nitrate 4-hydrate with a little amount of magnesium nitrate 6-hydrate was prepared as the oil phase to making the nanoemulsion. The presence of magnesium ion will induce the formation of  $\beta$ -TCP and will encourage the spreading and adhesivity of osteoblast cells. The aqueous solution of (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> with or without nanoclay suspension was prepared separately. The molar ratio of Ca/P was kept to 1.5 for TCP formation.

*Chemically treated Ti sample to induce apatite deposition.* The introduction of a thin bioactive TiO<sub>2</sub> film by H<sub>2</sub>O<sub>2</sub>-oxidation and hot water aging on Ti surface can in turn induce the formation of Ca-P layer on implants [7].

*Nanoemulsion coating on Ti samples.* The chemically treated Ti samples were dip coated by acetone/water nanoemulsion. The coated Ti substrates were sintered at 800°C for 2h to form the  $\beta$ -TCP phase.

The nanocoated Ti samples were characterized by SEM, TF-XRD and nano-scratch test. MC3T3-E1 osteoblast-like cells were cultured on nanocoated Ti and cell viability and morphological observations were performed.

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### III. RESULTS

Clay nanosheet can be mono-dispersed into  $\beta$ -TCP nanomatrix by nanoemulsion coating method as shown in Fig. 1 (b).

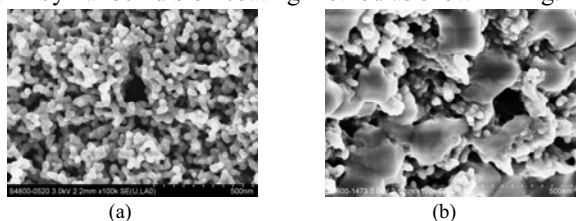


Fig. 1 Montmorillonite (MMT) nanosheet reinforced  $\beta$ -tricalcium phosphate (TCP) nanocomposite coatings on titanium: (a) pure TCP, (b) TCP/MMT nanocomposites (scale bar: 500 nm).

TF-XRD peak positions of nanocoating are in good agreement with JCPDS card of  $\beta$ -TCP (09-0169) as shown in Fig. 2.

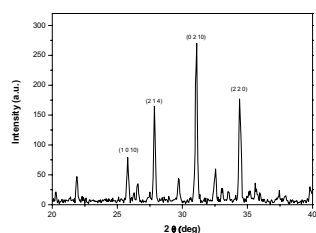


Fig. 2 TF-XRD pattern of nanocoating calcined at 800 °C.

A diamond indenter was used for face-forward scratching test using a NanoTest™ (Micro Materials). The depth variation of nano-coatings is shown in Fig. 3, which is as a function of displacement during nano-scratching. The result showed that the addition of nanoclay can dramatically improve the wear resistance of TCP coating. The wear resistance property is an important factor to evaluate the long-term service of implant materials. The fluctuation of the nano-scratch profile indicated the nanoporous features of the coating.

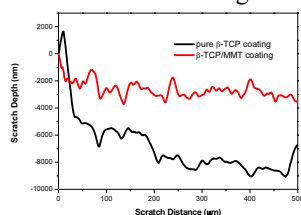


Fig. 3 Surface profiles of nano-coating with ramping normal load of 200 mN.

The cell viability was measured as optical density in different titanium surfaces which showed the good cytocompatibility of  $\beta$ -TCP/nanoclay coatings.

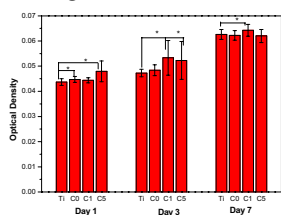


Fig. 4 Cell viability of osteoblastic-cells cultured on different titanium surfaces for 1, 3 and 7 days. Ti: un-coated, C0:  $\beta$ -TCP coated, C1:  $\beta$ -TCP/0.1wt%

MMT coated, C5:  $\beta$ -TCP/0.5wt% MMT coated. \*Significant differences between coated Ti and un-coated Ti ( $p < 0.05$ ).

The morphology of osteoblastic cells MC3T3-E1 after culturing for 1 and 4 days on Mg- $\beta$ -TCP nanocoated Ti substrates were shown in Fig. 5.

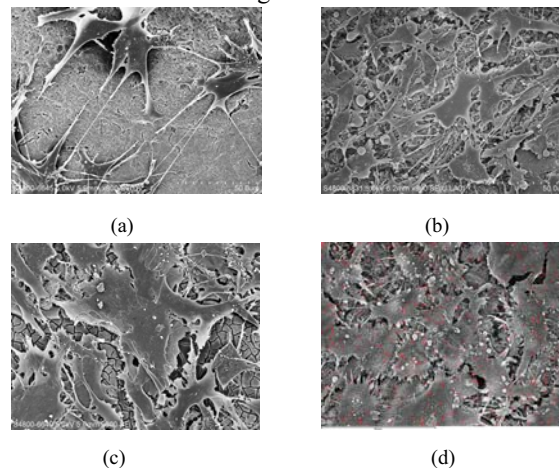


Fig. 5 SEM images showing MC3T3-E1 osteoblastic cell morphology after culture on (a) pure  $\beta$ -TCP, 1 day, (b) pure  $\beta$ -TCP, 4 days, (c) 3% Mg substituted  $\beta$ -TCP, 1 day and (d) 3% Mg substituted  $\beta$ -TCP, 4 days (the red dots are the calcium elemental mapping by EDX showing the biomineralization of bone cells), scale bar: 50  $\mu$ m.

### IV. CONCLUSIONS

A nanoemulsion coating was developed to coat nanoclay reinforced magnesium substituted  $\beta$ -TCP on titanium surface for enhancement the stability of orthodontic miniscrews by imparting nanoporosity and nanoroughness features on titanium surface.

### ACKNOWLEDGMENT

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