BioRemedi 2022

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Enhanced bioactivity and mechanical properties of commercially pure titanium sheets processed through Repetitive Corrugation and Straightening technique

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Abstract: Commercially pure titanium (Cp-Ti) is a significant biomaterial for load-bearing implant applications. However, it has lesser mechanical strength than titanium alloys, which needs to be addressed for its use as an orthopaedic implant material. Grain refinement of materials as per the Hall-Petch relation leads to the mechanical strengthening of metallic biomaterials. The severe plastic deformation method offers an opportunity for grain refinement of bulk materials to produce bulk nanostructured materials. In this study, Cp-Ti sheets were subjected to repetitive corrugation and straightening (RCS) technique for up to 4 passes. This study focused on the evaluation of the mechanical strength and *in-vitro* bioactivity of the processed samples. The RCS-processed samples were further characterized to evaluate their average grain size, wettability and protein adsorption properties. The reduction in grain size from 50 µm to 15 µm was observed after 4 passes in the optical micrographs. The hardness and tensile strength also increased proportionally for the processed sample. The reduced contact angle value ascertained the increase in hydrophilicity thereby enhancing the amount of protein adsorption due to the increase in surface energy of the material. The *in-vitro* bioactivity study of the RCS sample immersed in simulated body fluid (SBF) for 21 days resulted in dense hydroxyapatite formation as compared to the as-received samples. The Ca/P ratio of the apatite was found to be 1.67, which is equal to the stoichiometry of human bone. From this study, it can be concluded that RCS techniques result in enhanced bioactivity and mechanical strength of Cp-Ti sheets which can be used as medical implants.

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INTRODUCTION

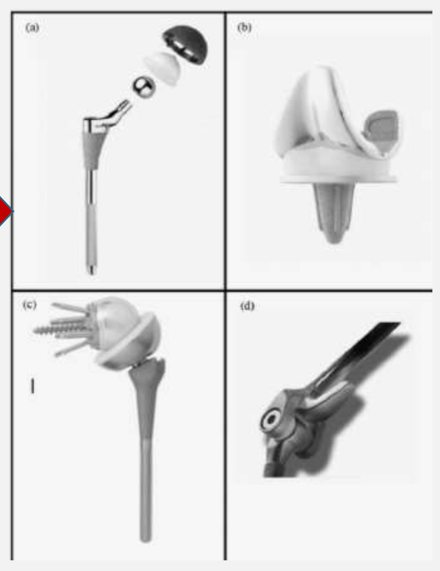
Biocompatibility

Low specific gravity

High specific strength

Corrosion resistance

Commercially pure titanium [Cp-Ti]



Load bearing implants

Less strength
Poor wear resistance
Bio-inert

solutions

Grain refinement

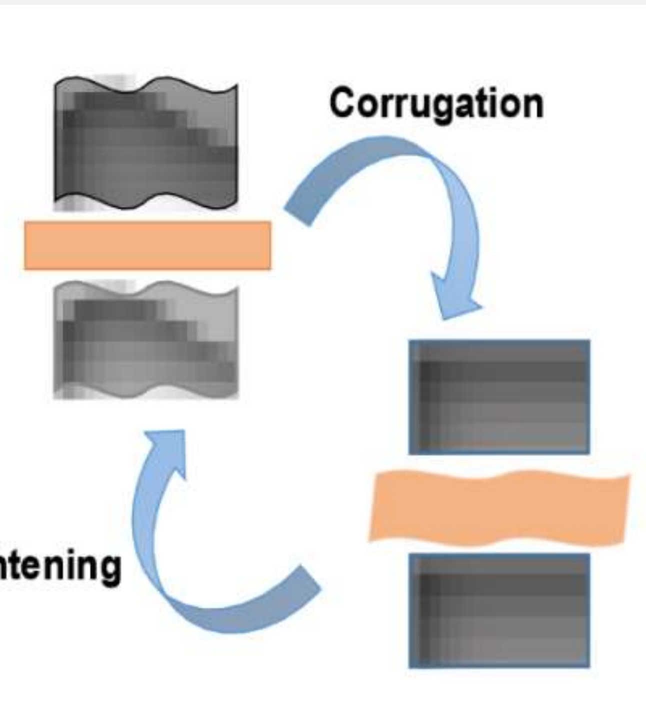
- Mechanical strengthening
- Bioactivity enhancement

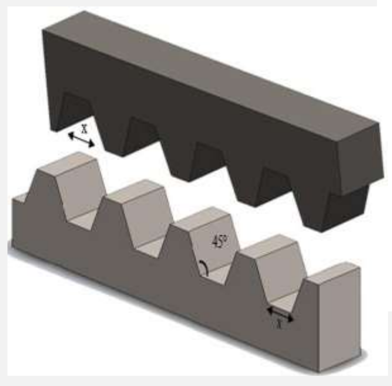
METHODOLOGY

Titanium sheets

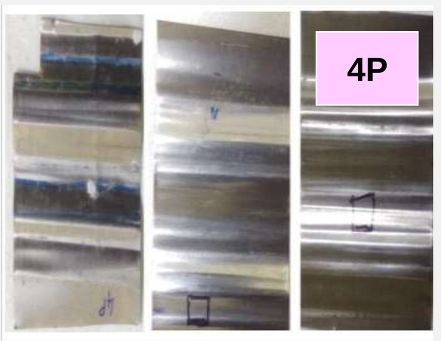
SPD techniques for grain refinement

RCS





RCS die design



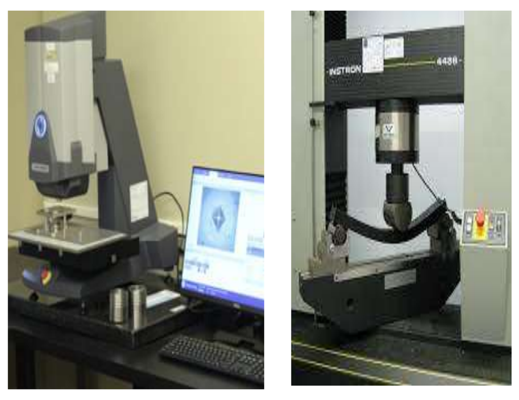
Processed samples

CHARACTERISATION AND EVALUATION

Optical microscope

Grain structure evaluation

Microhardness and Tensile



Mechanical strength evaluation

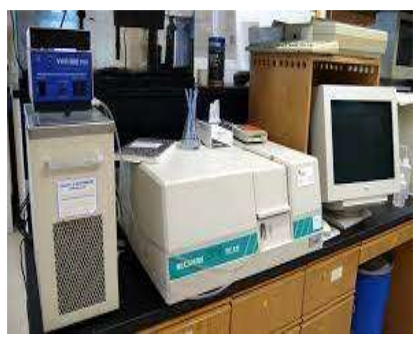
Contact angle

Wettability study

SEM

In-vitro bioactivity study

UV-VIS spectroscopy



Protein adsorption study

RESULTS AND DISCUSSIONS

Grain size analysis

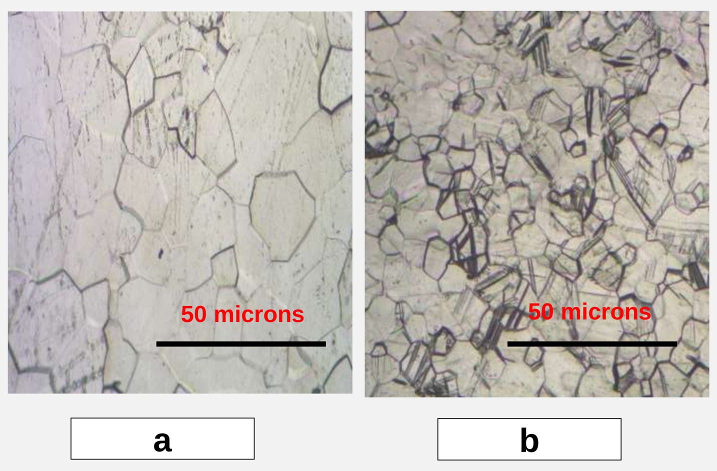


Fig.1. Optical micrographs of (a) As received [Cp-Ti As] and RCS processed sample [Processed Cp-Ti]

- The grain size of the commercially pure titanium as received sample (Cp-Ti) was 50 μm.
- The grain size of the Repetitive corrugation and straightening (RCS) processed samples, subjected to processing for up to 4 passes, was found to be 15 μm .
- Grain size reduction occurs which results in mechanical strengthening as per Hall-Petch relation.

Hardness and Tensile studies

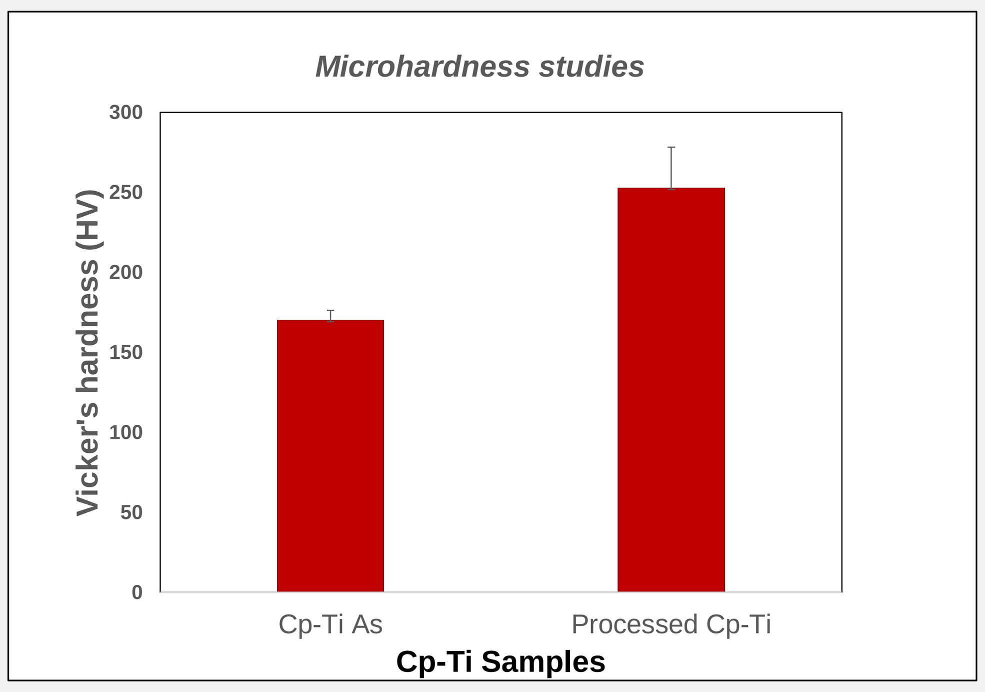


Fig. 2. Vicker's hardness of (a) As received [Cp-Ti As] and RCS processed sample (Processed Cp-Ti)

- The microhardness value of Cp-Ti As was found to be (171± 3.2) HV.
- The microhardness value of processed Cp-Ti was found as (252 ± 2.1) HV.
- The increase value of hardness indicates the mechanical strengthening of the sample.
- Table 1 shows the tensile strength of samples.
- The values reflects the mechanical strengthening of RCS processed samples mostly due to the strain hardening.

Table 1: Tensile strength of as received Cp-Ti and RCS processed samples

Sample names	Yield strength, σ_Y (MPa)	Ultimate tensile strength, σ_u (MPa)	% Elongation
Cp-Ti As	303.5 ± 2.4	410.3 ± 3.2	30.3 ± 2.1
Processed Cp-Ti	457.2 ± 3.7	498 ± 2.6	22.8 ± 1.4

Wettability studies

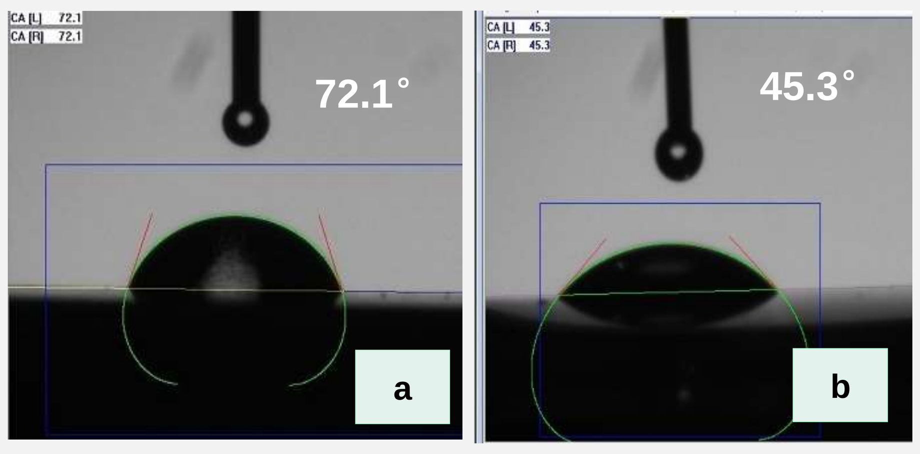


Fig.3. Static contact angle measurements of (a)As received[Cp-Ti As]and RCS processed sample [processed Cp-Ti]

- The contact angle measurement of Cp-Ti was found to be 72.1° whereas of RCSed sample was 54.3°.
- The decrease value indicates its increase rate of hydrophilicity, much needed for a biomaterial of orthopaedic applications.

In-vitro bioactivity

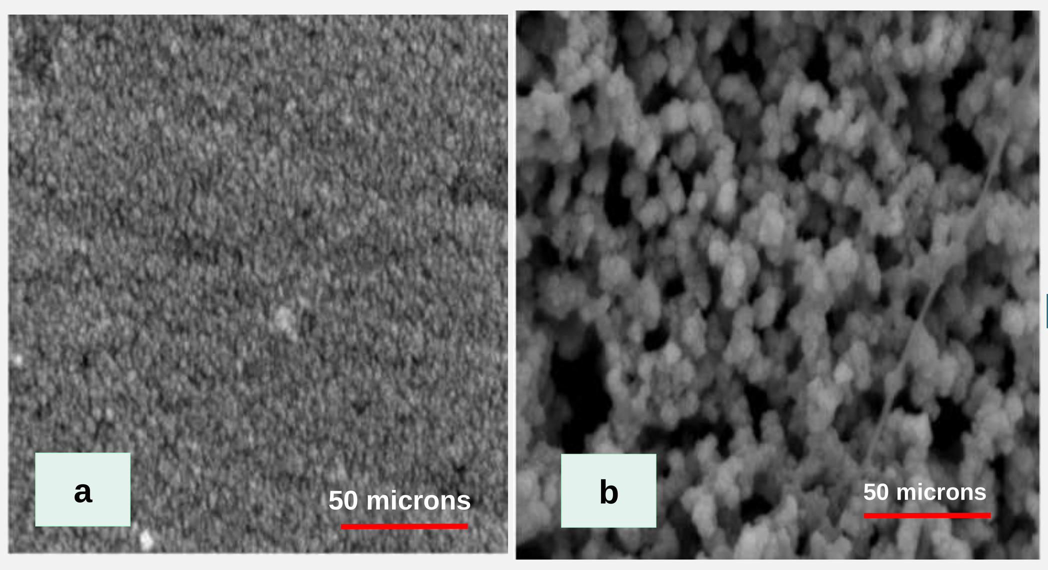


Fig.4. SEM micrographs of (samples immersed in SBF for 21 days) (a) As received [Cp-Ti As] and RCS processed sample [Processed Cp-Ti)

- The SEM micrographs were taken for the samples immersed in SBF solution for 21 days.
- A dense hydroxyapatite formation at the surface of RCSed sample was observed with a Ca/P ratio of 1.67 almost similar to stochiometric of human bone.
- This results in better osseointegration, enhanced adsorption and a faster wound healing for the load bearing applications.

Adsorption studies

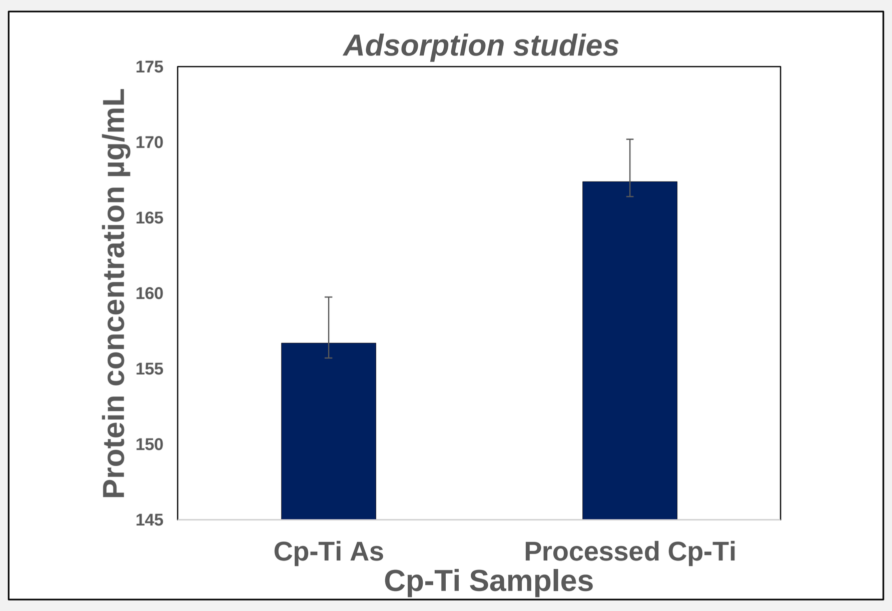


Fig. 5. Protein adsorption value of (a)as received [Cp-Ti As] and RCS processed sample [Processed Cp-Ti]

- Initially a standard calibrated BSA curve was drawn for calculation.
- The protein concentration was evaluated and for Cp-Ti it was 156.7 μg/mL and for processed Cp-Ti it was 167.3 μg/mL.
- The increase level in protein adsorption is related with the surface energy increased due to the lower contact angle.
- This enhances more protein adsorption, osteointegration and promotes faster wound healing.

Conclusion

- This study reported the effect of RCS performed on Cp-Ti sheets at room temperature. The experiment was carried out for 4 passes without encountering any cracks on the sample surface.
- The microstructural analysis confirmed the grain refinement and enhanced mechanical strength due to RCS technique. The results of contact angle and mechanical study showed the better results as per the hypothesised outcomes.
- The results of the *in-vitro* bioactivity study showed dense and homogenous apatite layer formation as observed in SEM analysis.
- It can be concluded that repetitive corrugation straightening method is one the state-of-the art techniques for Cp-Ti sheets that can be used in load bearing applications with assurance of improvement in mechanical strength as well as bioactivity of the implant material.

References

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