Synthesis and Processing of Hydroxyapatite Thin Films and Coatings for Translational Orthopaedic Surgical Implant Applications

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Introduction

Metal alloys such as Ti6C4Al has been widely used for internal trauma fixation of fractured bones by orthopedic surgeons due to their unique combination of high mechanical strength, corrosion resistance and biocompatibility.

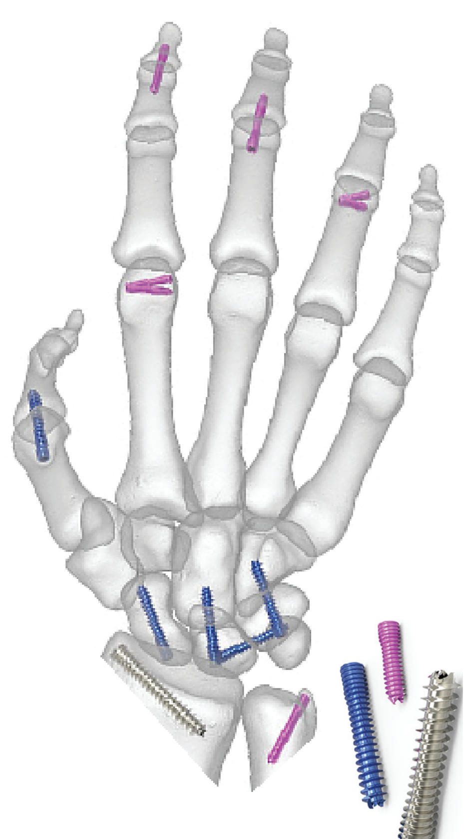
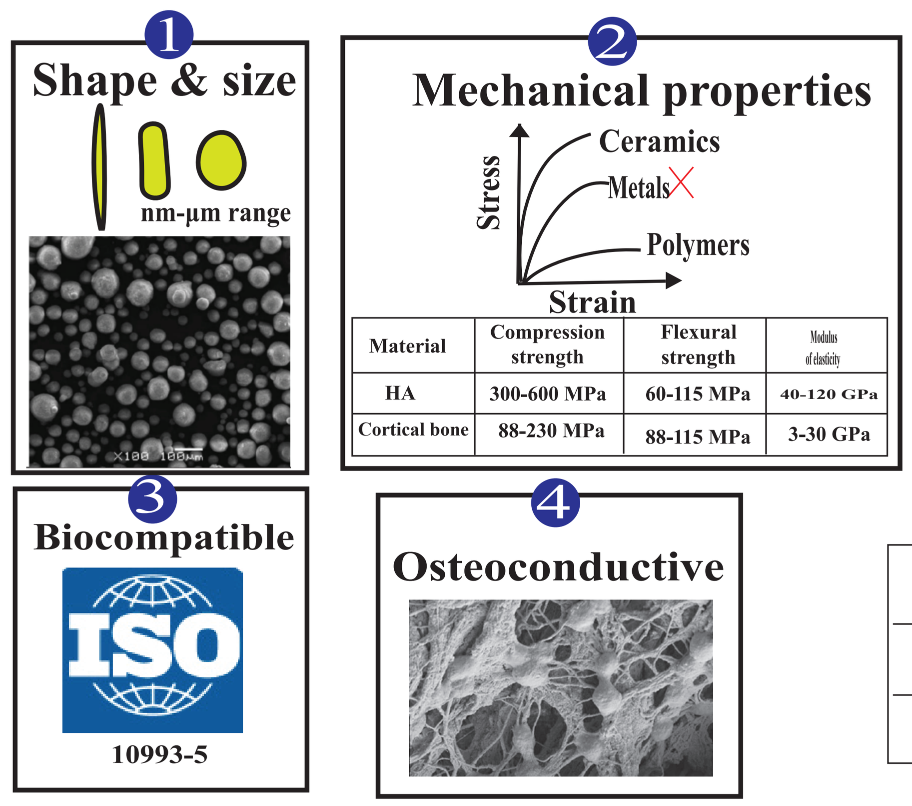


Fig 1: Internal fixation metallic hardware currently used in repairing bone fractures. Ref- Acumed

However there is an unmet clinical need for non-metallic next generation internal fixation devices. The advent of bioinspired design and advanced regenerative biomaterials has now made possible the exploration of advanced polymer-ceramic composites that can potentially replace rigid, metallic surgical hardware that continue to cause pain, infection, and migration.

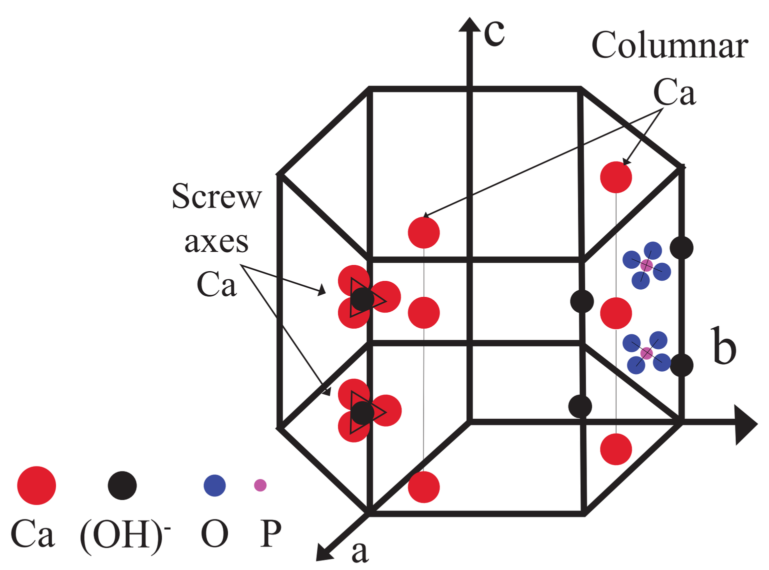
The objective of this project is the synthesis of hydroxyapatite (HA), the bioceramic mineral component of bone, by the sol-gel process as a means to synthesize conformal hydroxyapatite films and coatings.

A major advantge of sol-gel technology is its ability to produce extremely thin films and coatings of complex geometries. The goal of this research is to process HA thin films and coatings and evaluate its feasibility for fixation devices in the use of next generation internal fixation devices.



Material	Compression strength	Flexural strength	Modulus of elasticity
HA	300-600 MPa	60-115 MPa	40-120 GPa
Cortical bone	88-230 MPa	88-115 MPa	3-30 GPa

Fig 2: Physicochemical and Biological Properties of HA

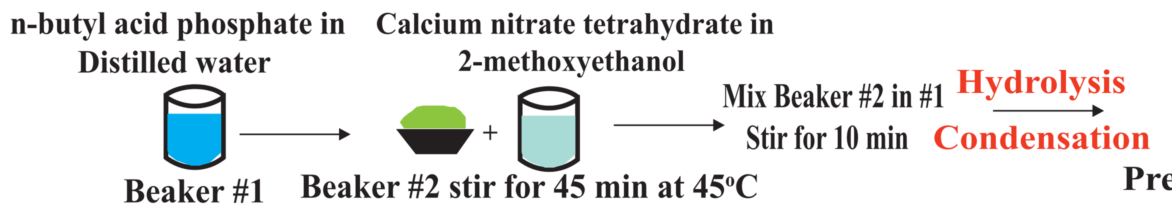


AXIS	Cortical bone	Hydroxyaptite
a-axis	9.419	9.422
c-axis	6.880	6.800

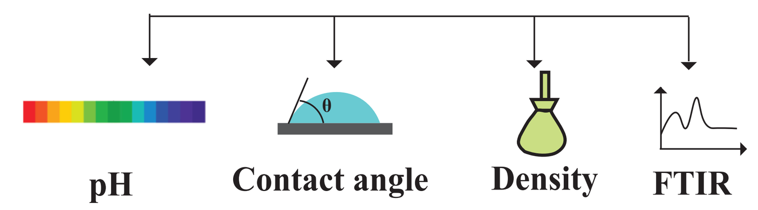
Fig 3: Unit cell of HA & Lattice parameter of HA vs Natural cortical bone

Methods

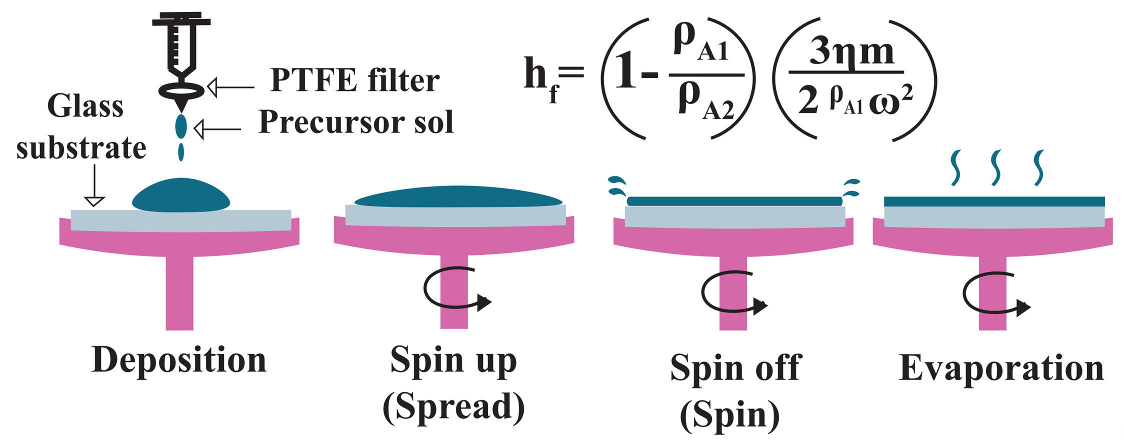
1.Formation of precursor sol



2. Characterization of sol

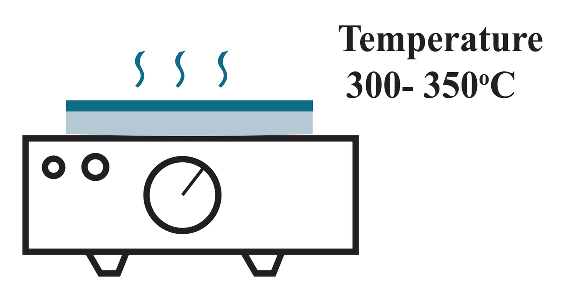


3. Preparation of thin films using spin coating technique

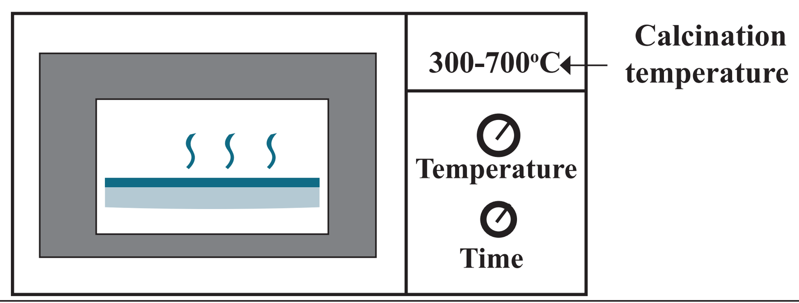


$$h_f = \left(1-\frac{\rho_{A1}}{\rho_{A2}}\right)\left(\frac{3\eta m}{2\rho_{A1}\omega^2}\right)$$

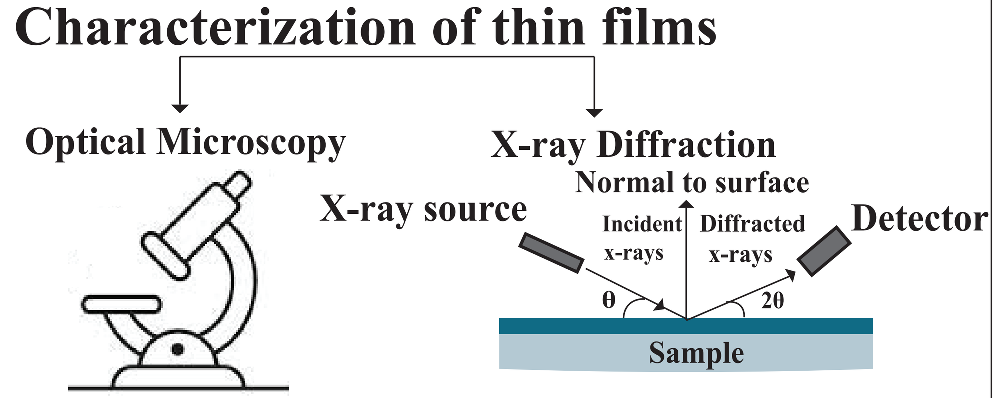
4. Drying thin films on hot plate



5. Calcination of thin films



6. Characterization of thin films



Results

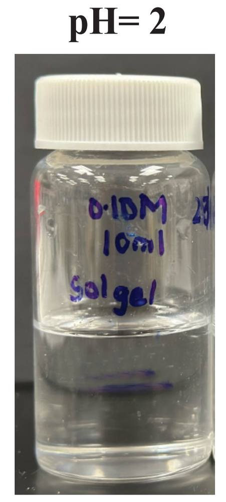


Fig 4: 0.10 M precursor sol

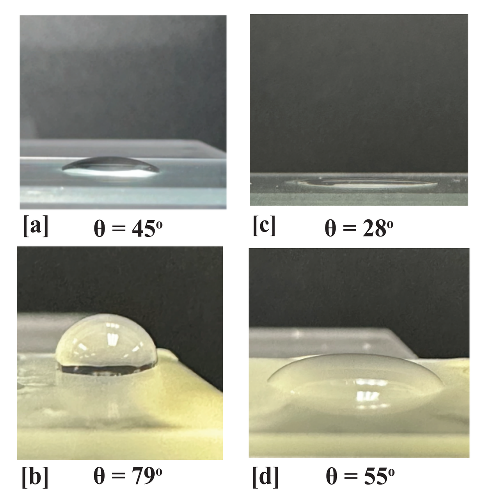


Fig 5: Contact angle measurement a) Water on glass b) Water on beeswax c) Sol on glass d) Sol on beeswax

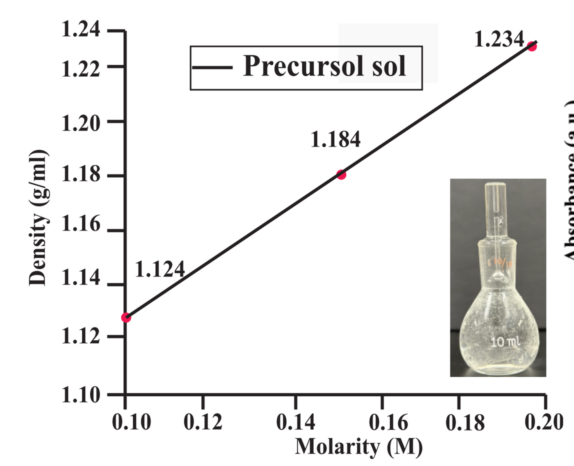


Fig 6: Density measurement of precursor sol

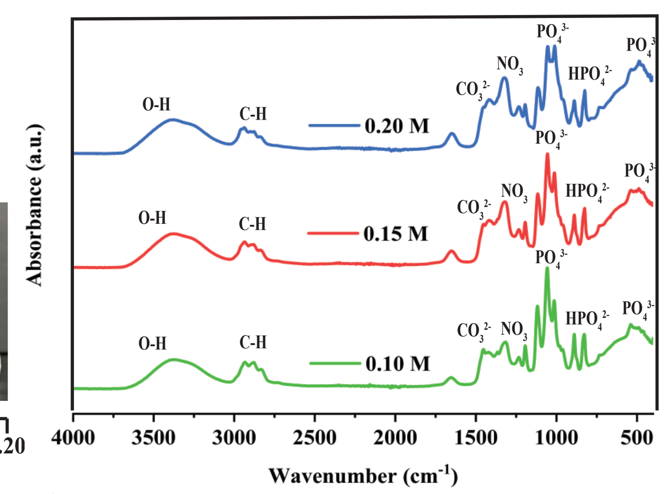


Fig 7: FTIR of precursol sol

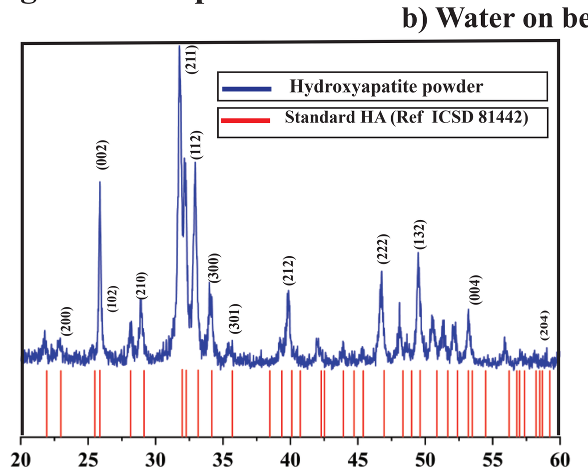


Fig 8: XRD spectra of HA powder

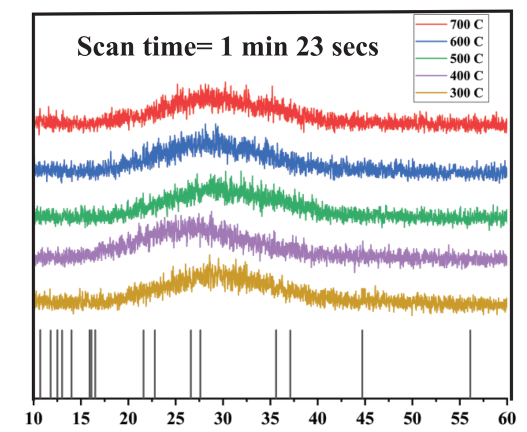


Fig 9: Preliminary XRD results of fired HA thin films

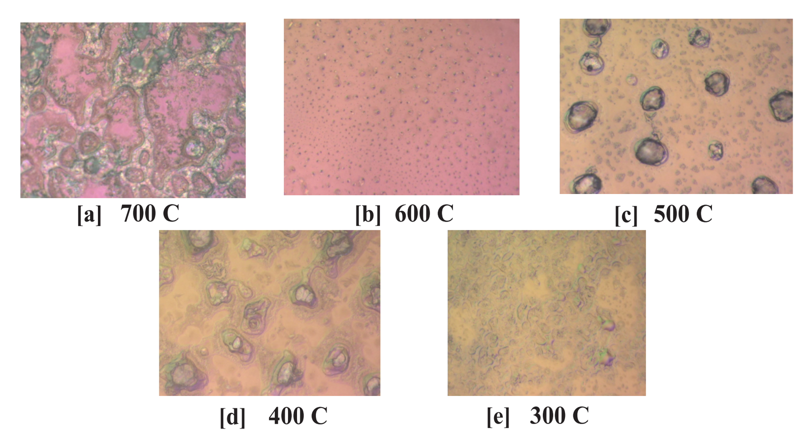


Fig 10: Optical micrographs of fired HA thin films at 200X

Discussion & Conclusion

HA precursor sols were successfully made. A key parameter to develop thin films and coatings was to determine the wettability of the sol as measured by contact angle. Contact angle results shown in fig 5 indicate that HA sol provides sufficient wetting on both glass and beeswax substrate ($\theta<90^{\circ}$). FTIR spectra shown in fig 6 verify the presence of HA precursors. XRD spectra of HA crystalline powders matched the HA standard card (fig 8). XRD spectra of sol-gel derived HA thin films (fig 9), shown in the corresponding optical micrographs (fig 10), fired at 300-700C did not result in peak formation primarily due to short scan time.

Next Steps

Develop sol gel dip coating and lost wax casting processes to create conformal HA internal fixation implant coatings.

Acknowledgments

The author is grateful for the support of Dr.Terry Alford and Dr. Olivia Burnsed at ASU, Dr. Kevin Dicker at Mayo Clinic of Arizona, Emmanuel Soignard and David Wright at Erying Materials Center at ASU.

The author is grateful for the support from all members of BioICAS lab at ASU.

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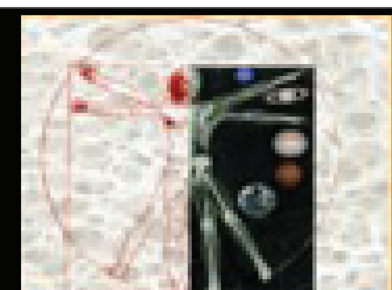

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