Automated Fabrication of Nonresorbable Bone Implants Using Laminated Object Manufacturing (LOM)

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[1]Master's thesis, Chemical Engineering, University of Dayton, May, 1998.

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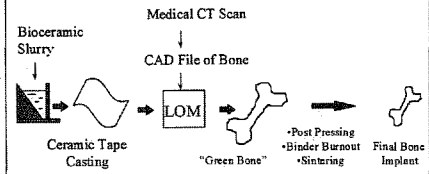
RPDL Rapid Prototype Development Laboratory

Objective

Demonstrate Feasibility of Using Ceramic Tapes with the LOM Process to Fabricate Functional Bioceramic Parts for Use as Bone Implants.

- Bioceramic Material Formulation
- Tape Casting
- LOM Process Development
- High Temperature Ceramic Post Processing
- Mechanical Testing

Bioceramic LOM Process Overview



Step 1: Material System Selection

Hydroxyapatite (HA) : $Ca_{10}(PO_4)_6(OH)_2$

- Proven biocompatibility
- Close resemblance to natural bone
- Used mostly for non-load-bearing applications
  - general repair of bone defects
  - augmentation of the alveolar ridge
  - middle ear reconstruction
  - plasma sprayed coating on metal implants
- ***However - HA is difficult to sinter!***

Material System Selection

- Composite system consisting of Hydroxyapatite and calcium phosphate glass was chosen. Calcium phosphate glass acts as a binding agent to hold the HA particles together
- Similar composite systems have been investigated
  - G. Graves et al — increased porosity
  - J.C. Knowles et al — improved mechanical strength

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LOM Trials

- Lamination trials were conducted with a 2"x 4" part divided into eight one-inch squares
- Adjusted tape binder formula to produce adequate lamination
- Once a suitable tape formulation was achieved a CT file of a wrist bone was used to make a 3-D bone part

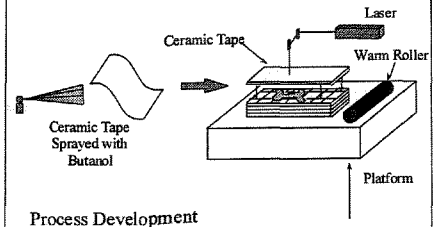
LOM Parameters

Roller Speed	5.08 cm/sec
Roller Temperature	25°C
Roller "Pressure"	0.635 mm
Laser Power	9 – 20 %
Laser Speed	12.7 cm/sec
Laminating Solvent	Butanol

Factors Involved in Lamination

- Type and amount of binder present in the tape
- Type and amount of solvent used for lamination
- Roller temperature, speed, and pressure

Step 4: LOM of Bioceramic Tapes



Process Development

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Step 5: High Temperature Ceramic Post Processing

- Pressing,
- Binder Burnout
- Sintering

Post pressing is used to improve layer fusion and to prevent bloating during binder burnout

Powder Bed Pressing Technique

Burnout and Sintering

Temperature (°C)	Time (hours)	Applied Pressure (MPa)	Apparatus
25 – 80	2	0.069 – 0.104	Corn Meal Bed/Press
80 – 120	12	0.069 – 0.104	Corn Meal Bed/Press
120 – 300	25	0	Furnace
300 – 500	13	0	Furnace
500 – 800	20	0	Furnace
800 – 1050	2.5	0	Furnace
1050 – 1050	5 minutes	0	Furnace
1050 – 20	free cool	0	Furnace

Bioceramic Wrist Bone Implants Made with LOM

Mechanical Test Results

Flexural Strength of Bone, Ceramics and LOM Samples

Bone	50 – 100 MPa [7,8]
HA	40 – 210 MPa [48]
TCP	90 – 154 MPa [48]
Bioglass	>120 MPa [48]
LOM Samples	18.8 MPa

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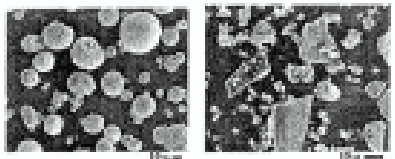
SEM of HA & Glass



HA particles, as received

glass, as received

Step 2: Material Formulation

Hydroxyapatite (HA) $Ca_{10}(PO_4)_6(OH)_2$ + Calcium Phosphate Glass

Combined in HA/glass ratios ranging from 10:1 to 1:1

- Pressed pellets for each ratio
- Sintered pellets using various cycles
- Measured compressive strength of pellets

Compression Test Results

(sintered pressed pellets)

	HA/glass ratio	Compressive Strength (MPa)
cycle 1 (1050°C, 5 min)[1]	10:1	81.6
	5:1	99.8
	3:1	104.8
cycle 2 (1050°C, 5 min)[2]	3:1	130.2
	1:1	73.2
cycle 3 (1050°C, 50 min)[2]	3:1	129.5
	1:1	83.3
cortical bone	--	100 - 230

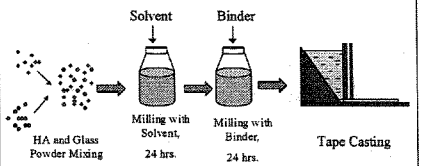
[1] rapid heat up cycle (sample placed in oven preheated at 1050 °C)

[2] heat up cycle = 5°C/min

Formulation Selection

- 3:1 ratio was selected based on compressive strength
- However, FTIR analysis of sintered specimens revealed the presence of a reaction by-product, Tricalcium phosphate (TCP), at a level of 10%. Because TCP at this level is resorbed in the human body, the material system cannot be considered nonresorbable in a strict medical sense.
- Despite the presence of TCP, the current 3:1 HA/glass system was adopted to serve as a model system for use on the LOM

Step 3: Tape Casting



Factors Involved in Tape Casting

- Slurry composition and viscosity
- Doctor blade setting
- Casting speed
- Drying conditions

Slurry Composition

Slurry Component	% of the total slurry weight	Role
HA	38 – 43	primary ceramic
Calcium phosphate glass	13 – 14	bonding ceramic
Menhaden fish oil	1 – 1.5	deflocculent
Ethyl Alcohol	9.3 – 10	slurry solvent
Trichloroethylene	24 – 25	slurry solvent
Poly vinyl butyral	2.7 – 4.5	primary binder
Poly ethylene glycol (MW 400)	2.9 – 4.8	plasticizer
Butyl benzyl phthalate	2.3 – 4.1	plasticizer
Stearic Acid	0 – 1.5	release agent

SEM of Tapes

HA/Glass Tape

HA/Glass Tape Binder Removed

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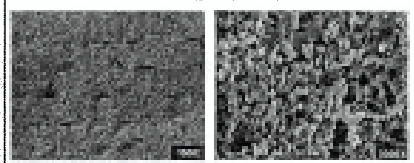
Other Properties of Sintered Bioceramic LOM Parts

- Compressive Strength = 30 MPa
- Specific Gravity = 2.77

Microstructure of Sintered LOM Part



Fracture Surface

Conclusions

- HA/glass formulation can be cast into useable tapes for lamination on the LOM
- HA/glass tapes are appropriate to form LOM shapes with acceptable adhesion between layers
- A 3-D bone shape was successfully produced from a CAD file taken from a medical CT Scan
- High temperature post-processing techniques were successfully used to densify the LOM bones without distorting the complex shape.

Conclusions (cont.)

- The mechanical properties of the specimens made with the best HA/glass formulation (3:1 ratio) were lower than that of cortical bone and pure sintered HA by a factor of two or three.
- The mechanical properties are expected to rise with optimization of the various processes involved, particularly post-pressing (elimination of interlaminar voids) and sintering.
- Overall feasibility of producing bioceramic bone shapes from LOM has been established.