The Use of Combustion Synthesis to Produce Porous Intermetallic Biomaterials

Matthew Karsh, Dr. Reed Ayers
Colorado School of Mines

Statement of Purpose: One of the primary issues facing the biomaterials industry is the high cost and difficulty to consistently manufacture materials used in implants. A production method under investigation to produce biomaterials is combustion or self-propagating high temperature synthesis (SHS). SHS allows for rapid processing of materials without any prolonged high temperature treatment. Synthesis occurs through a fast, self sustaining reaction driven by the exothermic heat release of the reaction. SHS reduces processing times and energy requirements, offers a high-purity final product with good control of the product attributes. This research will investigate and model how the SHS process can be used to produce dense and porous NiTi and CoCr intermetallics for the biomaterials industry. The ultimate goal is to produce NiTi that meets ASTM F-2063 specifications and CoCr that meets ASTM F-75 specifications.

Methods: Nickel, titanium, chromium and cobalt powders mixed to the designated equiatomic ratios are compacted into cylinders of various heights and green densities using a 0.5" diameter mold. Each cylinder is placed on a tungsten ignition coil in a tube furnace and preheated to a range of temperatures, 300° C - 400° C for NiTi and 400° C to 800 ° C for CoCr) in order to provide enough thermal energy for the SHS to take place. A current is passed through the coil; 42A and 20V for 3 seconds is used for the NiTi while a 50A current with 20V for 5 seconds is used with CoCr. After reaction, each sample is sectioned using a 4" cubic boron nitride wafering blade, mounted in bakelite and polished to a one micron finish. Materials are characterized using a variety of techniques including optical microscopy, scanning electron microscopy, X-ray diffraction and energy dispersive spectroscopy (EDS).

Results: It is possible to produce high-purity, porous NiTi and CoCr. Using a preheat of 45 minutes at 375° C to produce NiTi with porosities between 50-58%. This is slightly below the ultimate goal of 70% which is similar to trabecular bone. In regard to CoCr, a preheat of 1.75 hours at 775° C can produce monoliths with a final porosity of approximately 50%. Subsequent characterization of the materials using X-ray diffraction shows that SHS produces predominately equiatomic NiTi other intermetallics including Ni_3Ti, $NiTi_2$ and Ni_3Ti_4 are present. These intermetallics are desired as they affect the shape memory and superelastic properties[1]. However, most of the samples feature a majority of the NiTi phase. EDS analysis does show a higher level of oxygen and nitrogen then what is required in the ASTM standard.

Synthesis of CoCr has proven to be more difficult. A 0.3/0.7 Co to Cr ratio is used which is equivalent to the ratio prescribed in ASTM F-75. Due to its low exothermicity, the reaction requires a substantially higher preheat than NiTi. However, a CoCr product can be formed. The system is very susceptible to residual oxygen in the powders. The materials produced do not feature the desired porosity. The largest porosity to date generated is below 40%. Subsequent EDS analysis shows that each sample has a combined oxygen and nitrogen content of 5-10%. This may be the result of oxygen and nitrogen present in the argon process gas or through imperfect seals present in the experimental setup. The CoCr samples produced do not feature the desired porosity. The largest porosity generated to date is below 40%.

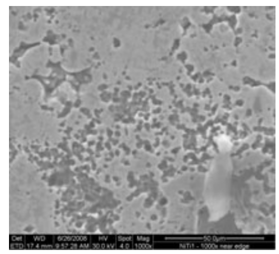

Figure 1: SEM of porous reacted NiTi.

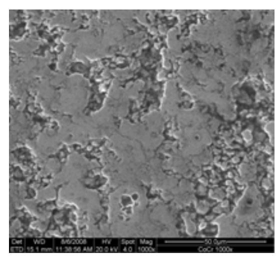

Figure 2: SEM of CoCr showing reacted and unreacted material.

Conclusions: It is shown that NiTi and CoCr can be synthesized by SHS. It is necessary however to continue to refine the processing techniques in order to obtain the required porosity and microstructure needed for bone implant applications. Characterization was able to determine the phases present in the materials and show that creating equiatomic NiTi and CoCr with an approximate .3/.7 ratio is possible. Future work will involve optimization of the SHS process to be able to produce samples with better purity and higher levels of porosity.

Reference: 1. Otsuka K. Prgs in Mtrl Sc. 2005:50(511-687)