

Introduction

Motivation:

- Nanostructured pure titanium (Ti) has the potential to launch a new generation of metals for medical implants, as nanostructuring enhances bulk, surface, and biological properties.
- The healing response of bone to titanium can be accelerated 20-fold by nanostructuring to reduce the metal's crystal size and increase grain boundary density. Nano-Ti is 50%-100% stronger than normal Ti, so much stronger that it can be more susceptible to microcracking.
- Nanostructuring Ti is done by subjecting the long rods to high shear deformation. One way to do this is by Continuous Equal Channel Angular Pressing (C-ECAP) and then swaging into long rods. These processes create residual stresses that must be removed by annealing.

Research Objectives:

- Determine how to incorporate the benefits of nano-structuring long rods and bars of titanium, into medical implants such as spinal rods or dental implants.
- Observe microcracks within the first 0.010 inches of the swaged surface.
- Evaluate the annealing response by measuring the microhardness.

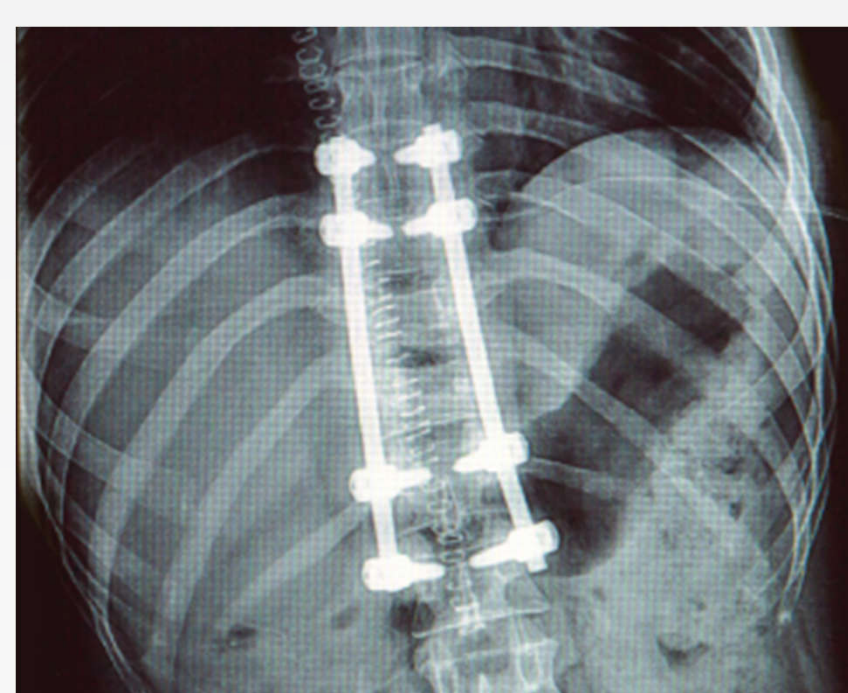
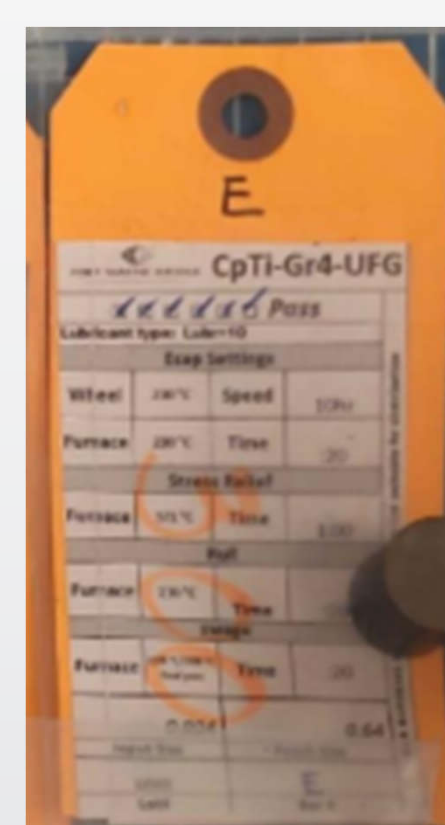


Figure 1: Titanium Spinal Rod Implant

Materials & Methods



Materials:

- 9 Ultrafine Grain Grade 4 Titanium 0.64" Round Bar Samples
- C-ECAP + RCS rolling + Stress Relief + Swaging to 0.64" + Post-Swaging Annealing
- 8 samples were subjected to 1 hour annealing treatments between 200°C and 375°C

Figure 2: Samples received in bags marked with processing information

Sample Prep:

- Examining transversely sectioned samples
 - Grind to 800 grit with Leco Auto-Grinder/Polisher
 - Polish with Buehler Vibromet II, Colloidal Silica Solution for 24 hours

Characterization:

- Optical Microscopy
 - Imaging cracks > 100 microns deep at circumference
- Vicker's Microhardness
 - Evaluate Vickers Microhardness at the center and on opposing sides of the circumference



Figure 3: Manual Vickers Microhardness Indenter

Results

How does nano-Ti respond to annealing?

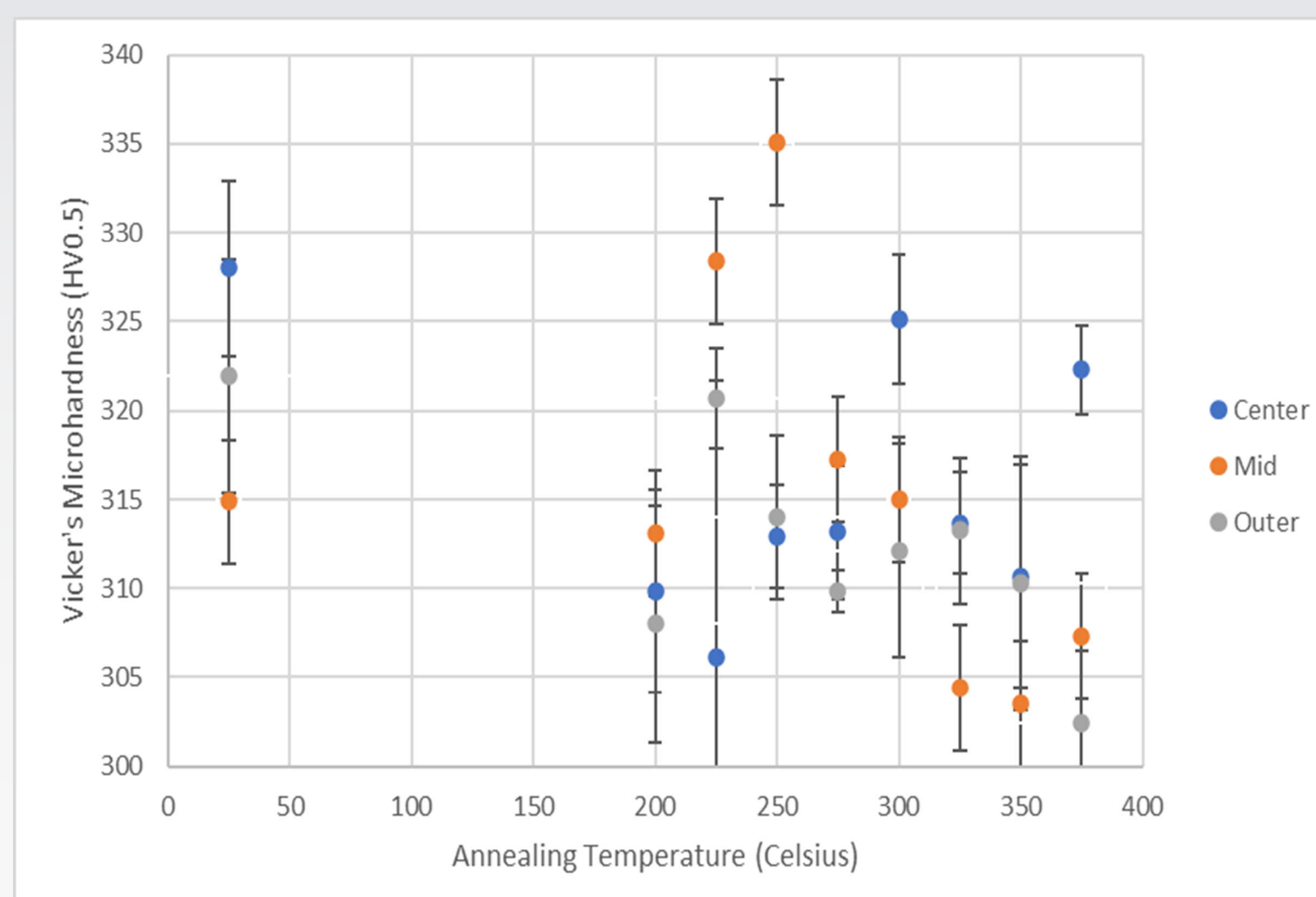


Figure 4: Plot of Annealing Temperature Versus Vickers Microhardness (HV0.5)

The annealing response to annealing is unique. Hardness first increases with temperature and before it decreases again. This is known as anomalous anneal hardening.

This phenomena occurs in pure nanostructured materials but is most prevalent in pure nanostructured titanium. Nanostructuring is applied to titanium because there is a need in the medical industry to have strong bio-inert metals. Pure nano-Ti doesn't need alloy elements that are needed to strengthen conventional alloys, and that are harmful to the human body.

Why does Anomalous Anneal Hardening happen?

There are two main theories as to why this occurs:

- Sub-boundary formation:** High shear deformation by C-ECAP processing induces extremely high dislocation densities.
 - Annealing at intermediate temperatures can cause dislocations to rearrange into tiny sub-boundaries.
 - These sub-boundaries are dense dislocation walls and can increase the strength of the metal during annealing.
 - Annealing at yet higher temperatures causes the boundaries sharpen and the dislocation walls to move, so the metal softens.
- 2nd Phase formation:** Pure titanium contains small amount of iron, and the iron forms clusters.
 - These iron clusters occur whether the material is annealed or not.
 - Annealing causes the iron to form into a second phase, called the beta phase.
 - This beta phase can increase the hardness of the material via iron diffusion during annealing.

Are deformation-induced crack small enough to easily remove?

The nanostructuring creates high strength material, partly due to increasing the high dislocation density. Super strong metals are more susceptible to surface cracking, which must be small enough to be removed easily. We found few cracks deeper than 0.010", so that they could be readily removed by centerless grinding.



Figure 5: 0.009" deep crack in nanostructured Titanium annealed at 375°C



Figure 6: 0.005" deep crack in nanostructured Titanium annealed at 300°C

Conclusions

To conclude, four major observations were made from the results of the Vickers microhardness and crack investigation:

- Samples confirm "annealing hardening" phenomena
 - Unique to nanostructured metals
 - Generally, annealing reduces hardness and strength of pure metals of pure metals. In nanostructuring, it can increase the strength.
- It is determined that the optimum annealing temperature was 275°C
 - This temperature shows the peak hardness, as well as being the beginning of the anneal hardening regime.
- The average microhardness was 314.52 +/- 36.22 HV0.5.
 - This is to be expected with the nanostructure and the post processing annealing.
- Cracks were present at the surface in three sections of swaged Nano-Ti 0.64" diameter bar
 - Only one crack exceeded a depth that would be removed by conventional centerless grinding of 0.010" (found at 0.017" depth)

Future Work

Future analysis will be conducted in two main areas:

- Anneal Hardening
 - TEM analysis could be used to show formation of dislocation sub-boundaries.
 - SEM analysis could be used to show growth of the iron clusters.
- Cracking
 - The processes of swaging and C-ECAP cause gradient in hardness, so the outside of the bar has higher hardness.
 - Further analysis could be done to evaluate the effects of surface annealing between C-ECAP passes. Rapid induction annealing could soften the outer layer and reduce over hardening of the surface of the material.
 - Analysis could also be done to evaluate the effects of induction surface annealing in between size changes for swaging.