Characterization of Radiopaque High Strength Alloys for Future Generation Implants

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Abstract: Next generation medical alloys aim to meet evolving biocompatibility and safety standards, requiring extensive testing and characterization prior to implementation. Fort Wayne Metals is seeking to gain insight into the viability of FeMnPt based alloys for radiopaque vascular implants. This study investigates and characterizes the corrosion behavior, mechanical properties, and microstructural characteristics of FeMnPt alloy samples in comparison to common medical alloys including 316 LVM stainless steel, 35N LT™, and L605.

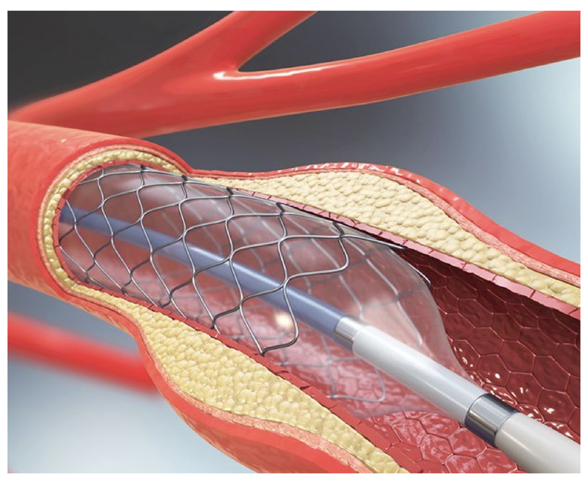
This work is sponsored by Fort Wayne Metals, Fort Wayne, IN

FORT WAYNE METALS

Background

Motivation: Cobalt based alloys including 35N LT™ and L605 have been in use for vascular implants for over seven decades. However, in 2017 the European Union Committee for Risk Assessment classified cobalt as a category 1B Health Hazard. Additional concerns due to nickel allergies have prompted the need for next generation alloys that meet evolving biocompatibility standards.

Implant Requirements: Vascular implant materials must meet specific requirements to function properly. Materials used in Balloon Type implants require high ductility, strength, biocompatibility, and high radiopacity. Corrosion resistance is dependent on the intended use of the implant. Permanent implants that will not require eventual removal, require long term corrosion resistance. Temporary implants however are designed to degrade at a controlled rate within the body to remove the need for a secondary potentially dangerous surgery for removal.



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Project Objectives: The focal objectives of this project include designing characterization experiments and recommending processing methods to optimize balloon-expanding and self-expanding devices. Characterization techniques are used to investigate the correlation between microstructure and functional performance in candidate medical device alloys. The corrosion performance of candidate alloys will also be investigated to determine the best potential application.

Experimental Methods

Sample Preparation: FeMn38Pt ingot samples were cut with a LECO VC-50 diamond saw and mounted using a LECO PR-4x Mounting Press. Wire samples (316 LVM, 35 NLT, FeMn28Pt, FeMn38Pt, and L605) were epoxy mounted in 1-inch cups with a 5:1 resin-to-hardener ratio. Samples were polished using a LECO Spectrum System 1000 – from 600 grit or 320 grit (FeMn38Pt ingot) to 1 µm diamond paste.

Optical Microscopy: Olympus SZX7 and Olympus BX41M microscopes were used to capture micrographs during and after polishing to evaluate surface uniformity, defects, and microstructure.

Scanning Electron Microscopy (SEM) & Energy Dispersive Spectroscopy (EDS): The Phenom Desktop and Apreo SEM captured high resolution images of the ingot and wire samples. EDS identified variances in elemental composition of the samples.

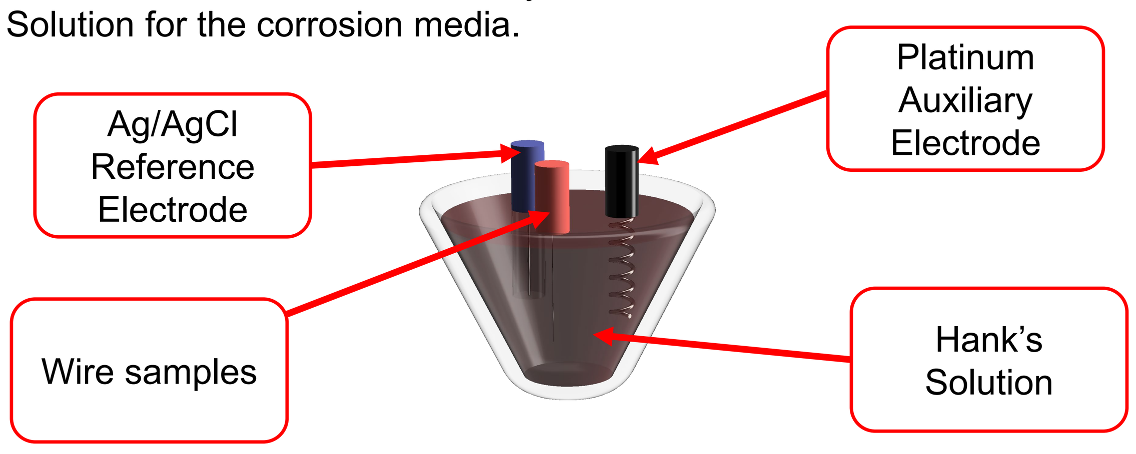
Mechanical Testing (Hardness): The Vickers hardness of the as cast FeMn38Pt ingot was tested using a Wilson Tukon 1202 Knoop/Vickers Hardness Tester. The sample was sectioned and polished prior to testing.

Mechanical Testing (Tensile Testing): Each of the wire samples were tested to determine the young's modulus, yield strength, and ultimate tensile strength. The samples were tested using a Admet eXpert 7600 Single Column Universal Testing Machine.

Heat Treatment: Wire samples underwent heat treatment at 450 °C and 650 °C for 1 hour in Thermocline furnaces, followed by air cooling and water quenching to assess microstructural changes.

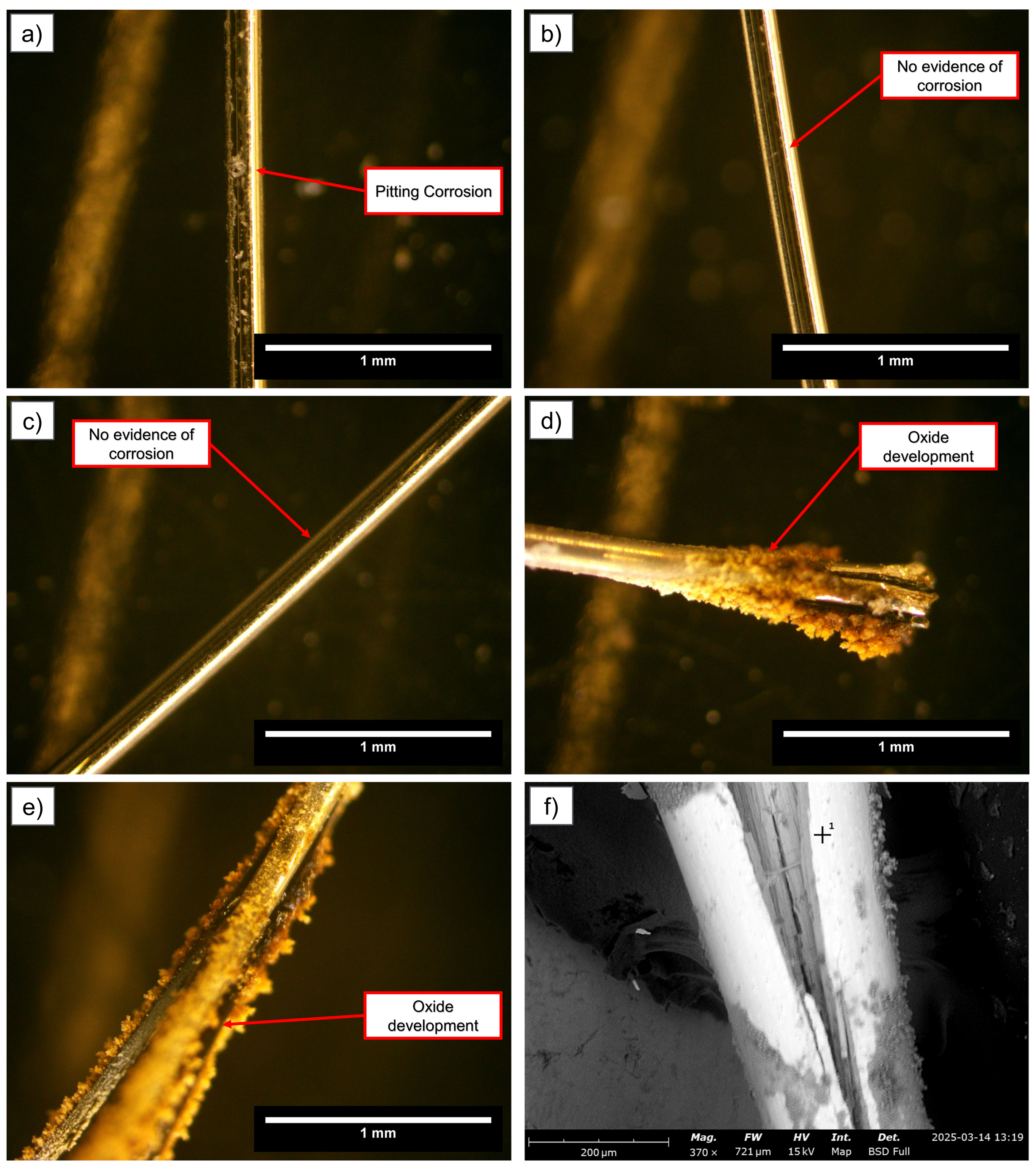
In Vitro Corrosion Analysis: Each of the wire samples were cleaned via sonification in acetone and deionized water prior to immersion in Hank's Solution. Pre-Corrosion masses were collected to calculate potential mass loss at 2-week intervals up to 6 weeks.

Cyclic Potentiodynamic Polarization: Each of the wire samples and cell were cleaned prior to experimentation. Measurements were taken using the MultiPalmSens4 Potentiostat, a BASi Ag/AgCl reference electrode, BASi Platinum auxiliary electrode, and Hank's Balanced Salt Solution for the corrosion media.



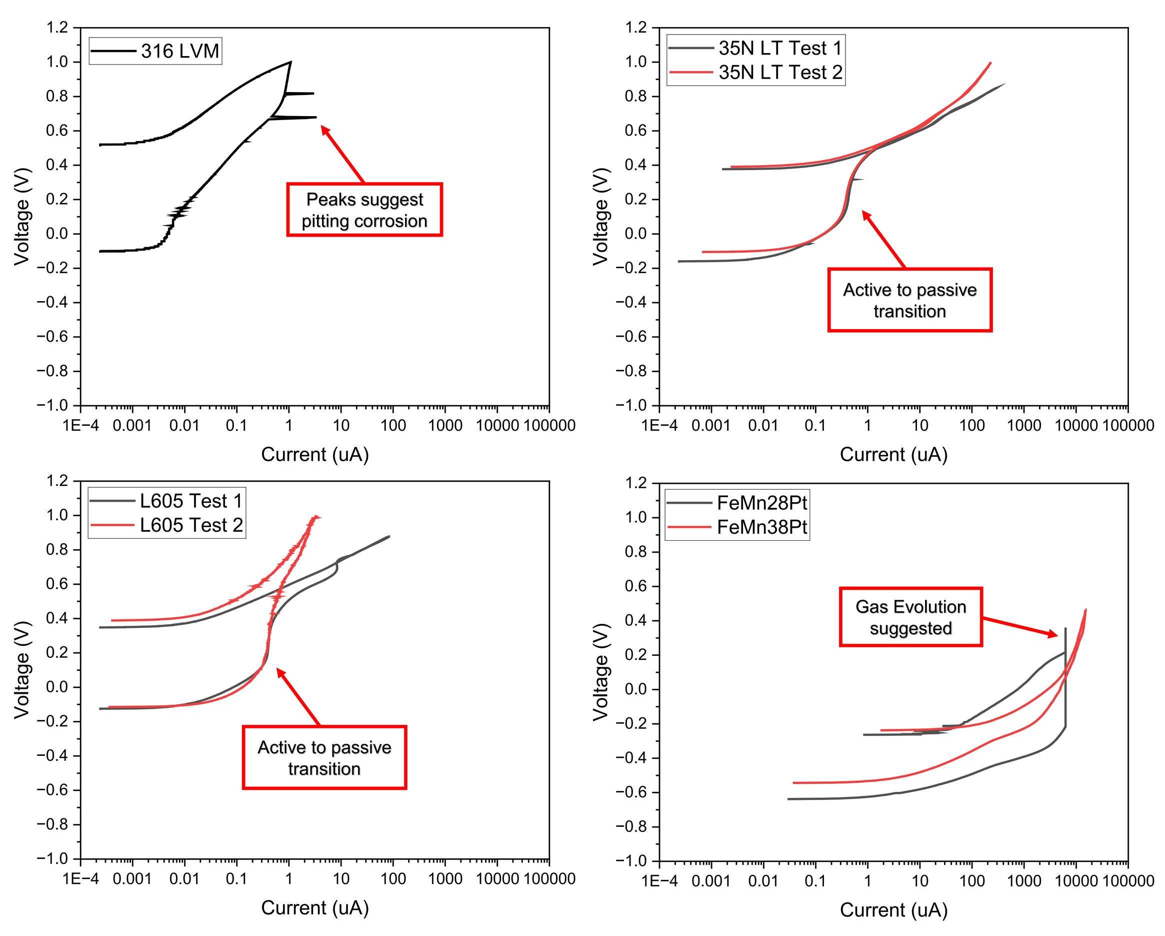
Corrosion Analysis

In Vitro Corrosion Analysis:



Wire samples after 4 weeks immersed in Hank's solution prior to cleaning are shown above. FeMnPt samples exhibit stress corrosion cracking at wire ends. a) 316 LVM Stainless Steel, b) 35N LT™, c) L605, d) FeMn28Pt, e) FeMn38Pt, f) FeMn38Pt SEM of crack

Cyclic Potentiodynamic Polarization (ASTM F2129-19):

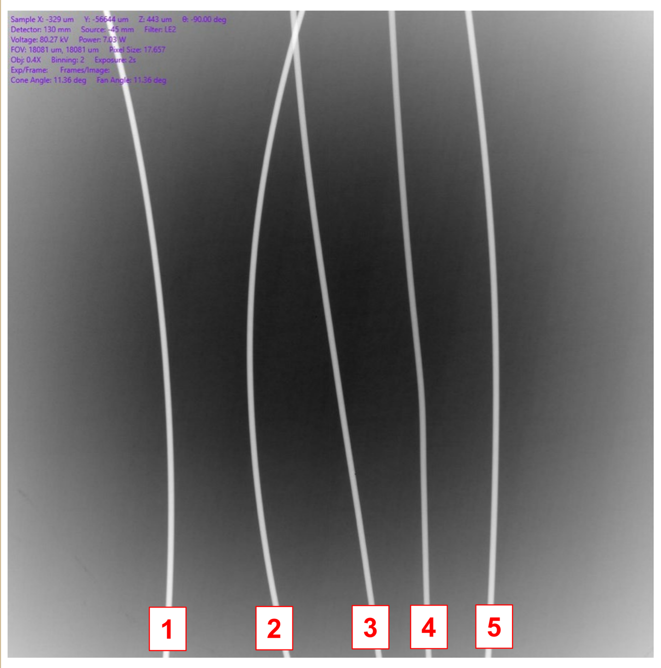


Anodic dissolution present in FeMnPt alloys:

$$Fe \rightarrow Fe^{2+} + 2e^-$$
$$Fe^{2+} + 2H_2O \rightarrow Fe(OH)_2 + 2H^+$$

$$Mn \rightarrow Mn^{2+} + 2e^-$$
$$Mn^{2+} + 2H_2O \rightarrow Mn(OH)_2 + 2H^+$$

Radiopacity



2D x-ray projections performed using the Zeiss Xradia 510 Versa - 3D X-Ray Microscope (3D XRM) at 0.4x optical magnification and a pixel-size of 17.657 microns. 1) Fe-Mn-38Pt, 2) Fe-Mn-28Pt, 3) 35N LT™, 4) 316 LVM, 5) L605

The FeMn28Pt, FeMn38Pt, and L605 samples display more radiopaque behavior than the 35N LT™ and 316 LVM stainless steel.

Alloy Comparison

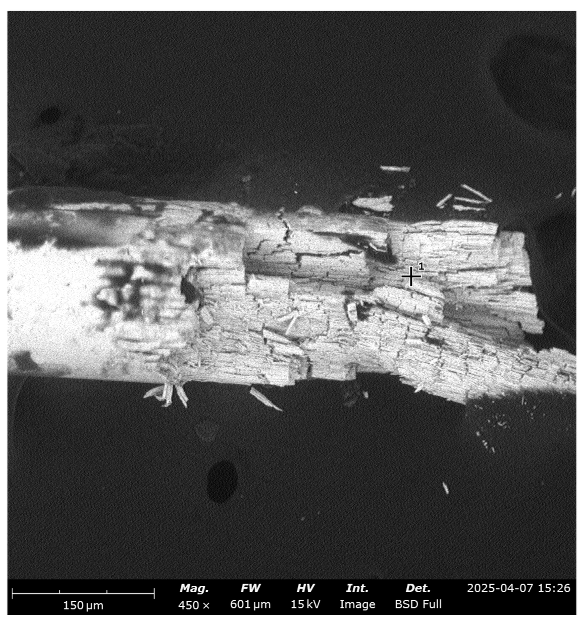
Mechanical Properties:

Alloy	Modulus of Elasticity (GPa)	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	% Elongation
316 LVM	32	1485	1732	5.1
35N LT™	57	1458	1730	5.2
L605	56	1516	2023	7.7
FeMn28Pt	55	1537	1911	4.4
FeMn38Pt	52	1547	1853	4.8

- FeMnPt alloys exhibit relatively similar mechanical properties compared to 316 LVM, 35N LT™, and L605
- However, the FeMn28Pt displays slightly higher Modulus of Elasticity and Ultimate Tensile Strength over the FeMn38Pt

Corrosion Properties:

- After In Vitro corrosion analysis, both FeMnPt samples displayed higher mass loss post corrosion than control alloys
- During Cyclic Potentiodynamic Polarization the FeMnPt alloys caused the Phenol Red in the HBSS to turn yellow around the electrode indicating a pH of at least 6.8 or lower
- Additionally, during imaging after corrosion, the FeMnPt samples effectively disintegrated as seen in the following image



Recommendations

There are several considerations that can be recommended for future integration of the Fe-Mn-Pt alloy samples. The first recommendation is to reanalyze the drawing speed of the wire samples due to evidence of microcracks being found in wire samples prior to experimentation. Drawing speed can impact the presence of microcracking in wire samples. It is important to understand how each material in the biomedical alloys behave when drawn. The second recommendation is to consider annealing the wire samples post-drawing to reduce internal stresses present. Several of the wire samples including the 316 LVM, Fe-Mn-28Pt, and Fe-Mn-38Pt experienced optimized mechanical behavior when heated at 450 °C for 1 hour.

Given the current form, the Fe-Mn-Pt alloys show use for temporary vascular implants. Assuming biocompatible breakdown, the alloys should dissolve safely in the body, avoiding the risks associated with secondary surgical removal. If long-term use is intended, further alloy development is necessary to ensure sustained mechanical properties and corrosion resistance. However, as seen during Cyclic Potentiodynamic Polarization the alloy suffers from severe degradation of mechanical properties often crumbling upon removal from the cell.

It is also recommended that the potential health effects of the FeMnPt alloys during degradation be investigated. As previously stated, the Hank's Balanced Salt Solution which includes the pH indicator Phenol Red changed to yellow around and below the FeMnPt electrode. This indicates a significant change in pH, which could be harmful to potential patients.

Acknowledgements

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