PRIME

Assessment of component and tubing performance made of Plasma-Arc-Melted Gen II material from Fort Wayne Metals

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Introduction

In the medical device industry Nitinol is a main material for various applications like stents and heart valve frames that are made from Nitinol tubing. The number of Nitinol ingot suppliers is limited and Fort Wayne Metals extended their melting capacity to supply Nitinol bar for tube manufacturing.

Consequently, the cooperative project PRIME (PRoficient Ingot Material Evaluation) was started driven by a collective of industry leaders along the process chain. The collective consists of the melt supplier Fort Wayne Metals, continuing along the process chain with Nitinol tube manufacturers Vascotube and Euroflex, and finally medical device contract manufacturers MeKo and Admedes. This collaborative project is dedicated to evaluate a new Nitinol source for tube processability and device manufacturing requirements.

The project includes different melting methods resulting in different material grades. This part of the whitepaper focused on so called Gen II material produced from Plasma-Arc-Melted Nitinol.

Keywords: Nitinol, PAM, Tube, Component

1 INGOT AND WROUGHT BAR

At Fort Wayne Metals (FWM) so called Gen II material was produced during early development of Altus™ Nitinol with plasma-arc melting (PAM) technology. Multiple bars from two different ingots (heat T-3246D6 and T-4684D6) were used for the following investigations that met the full requirements of ASTM F2063. A summary of the chemical requirements and analysis is shown in Table 1.

Element in % (mass/mass)	ASTM F2063	T-3246D6	T-4684D6
Ni	54.5 to 57.0	56.1	56.0
C, max.	0.040	0.0040	0.0030
Co, max.	0.050	<0.0030	0.0080
Cu, max.	0.010	0.0050	0.0090
Cr, max.	0.010	<0.0030	<0.0030
H, max.	0.005	<0.0010	0.0010
Fe, max.	0.050	0.0090	0.0080
Nb, max.	0.025	<0.0030	<0.0030
N, max.	0.005	0.0020	0.0030
O, max.	0.040	0.0170	0.0220

Table 1: Chemical composition requirements and analysis

Figure 1 is the differential scanning calorimetry (DSC) curves of the supplied FWM bars tested in the fully annealed condition in accordance to ASTM F2004. Heat T-3246D6 exhibited an Austenitic start (A_s) and finish (A_f) transformation temperature of -25°C and - 10°C respectively. Heat T-4684D6 had A_s transformation temperatures of -35°C and -29°C and A_f transformation temperatures of -24°C. The new Gen II Nitinol produced by FWM was able to meet the A_s transformation temperature range requirement of ASTM F2063 specifying a maximum range of 20 °C.

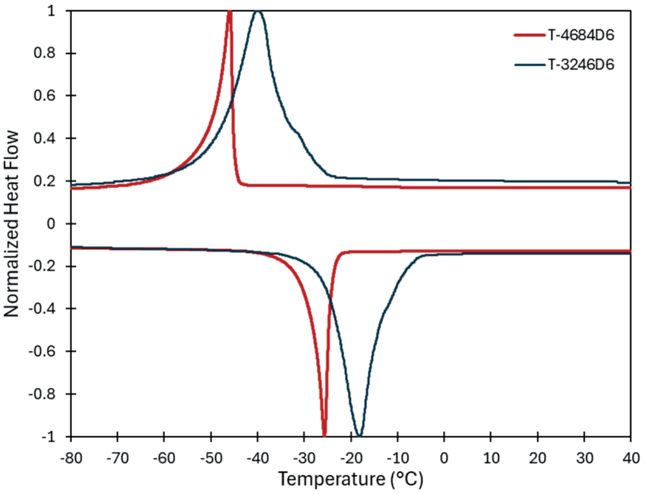


Fig 1: Differential scanning calorimetry of the supplied bars from FWM showing the fully annealed transformation temperatures.

Figure 2 is a representative micrograph of the supplied bar in annealed condition showing the grain size satisfied the ASTM F2063 requirement. Table 2 summarizes the grain size numbers.

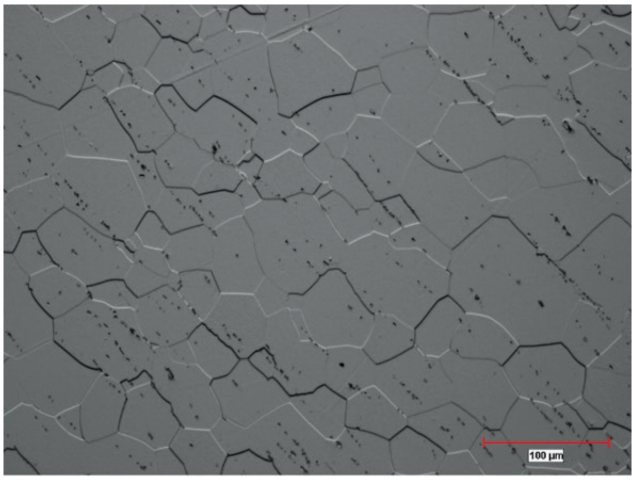


Fig 2: Representative micrograph of the supplied bar (T-4684D6) from FWM showing the grain size met the requirements of ASTM F2063.

	Grain size number
ASTM F2063	4 or larger
Heat T-3246D6	6.5
Heat T-4684D6	5.0

Table 2: Grain size requirement and analysis

Figure 3 shows representative non-metallic inclusions of the mill products. The ingots and mill products meet the PRIME Gen II specification for maximum inclusion size and area fraction as listed in Table 3. The more recently developed Altus™ Nitinol will be able to hold a max area fraction of 1.2% or better.

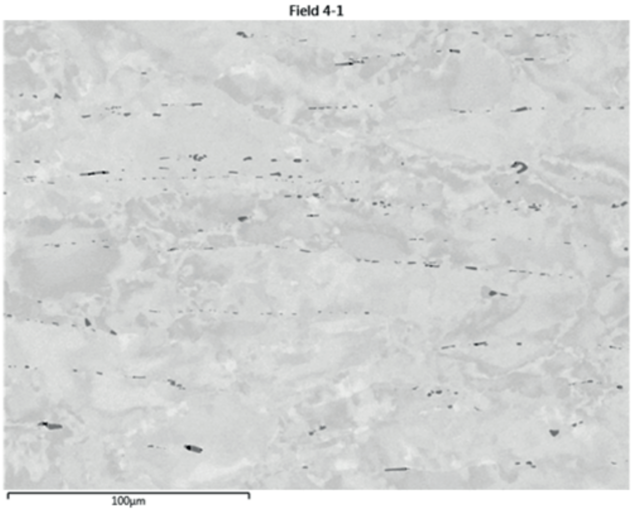


Fig. 3: Inclusion analysis of FWM mill product (T-4684D6) using 500x magnification

	Max. inclusion	Max. area fraction
Gen II specification*	20.0 µm	1.5 %
Heat T-3246D6	16.6 µm	1.0 %
Heat T-4684D6	19.9 µm	1.4 %

Table 3: Inclusion size of FWM Gen II ingots
*Altus™ Nitinol has a max area fraction of 1.2%.

Figure 4 is a representative tensile curve for the supplied FWM bars in the fully annealed condition. The tensile strength and elongation exceeded the 550 MPa and 15% minimum requirements of ASTM F2063.

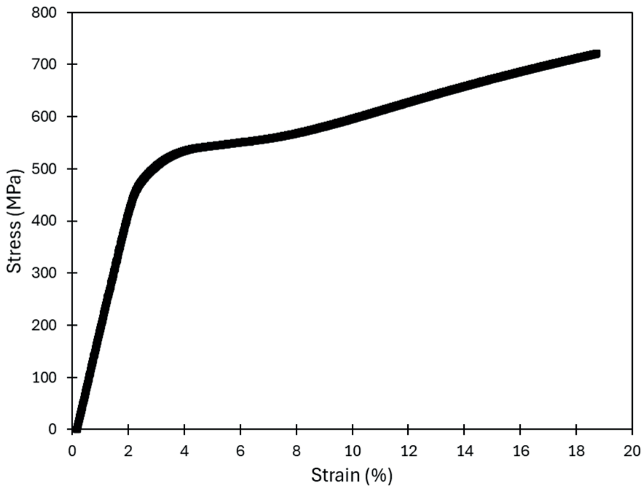


Fig. 4: Representative tensile test of the supplied bar from FWM showing that the fully annealed tensile strength and elongation met the requirements of ASTM F2063.

2 TUBE MANUFACTURING

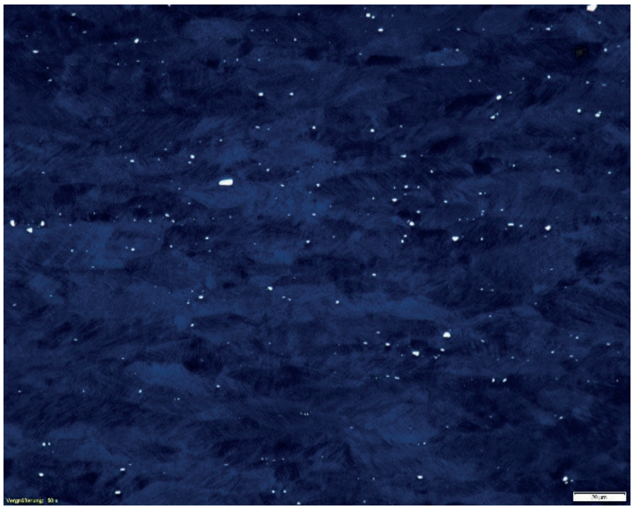
Tube manufacturer Euroflex and Vascotube manufactured two different tube sizes of Gen II Nitinol according to standard tube specifications. This report focuses on the tube size with outer diameter of 7.00 mm and wall thickness of 0.50 mm, in the following named 7x0.5. An extract of the standard tube specification is shown in Table 4.

Outer diameter	7.00 mm ± 0.025 mm
Wall thickness	0.50 mm ± 0.025 mm
Tensile strength	≥ 1100 MPa
Upper plateau strength (at 3 % strain)	≥ 380 MPa
Elongation at fracture	≥ 12 %
Residual elongation after 6 % strain	≤ 0.3 %
A_f temperature (crush method)	≤ 15 °C

Table 4 : Extract of tube specification. Mechanical properties tested according to ASTM F2516 at 22 °C

Each manufactured tube batch was divided and shipped to medical device contract manufacturers Admedes and Meko. Both medical device contract manufacturers conducted an extended incoming inspection by examining the microstructure and mechanical properties on the tube level of each batch.

Figure 5 shows metallographic cross-sections in the longitudinal direction of the finished tubing.



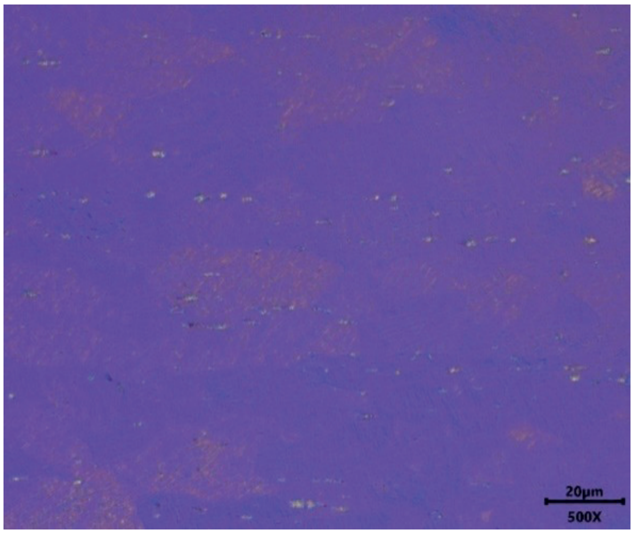


Fig. 5: Inclusion analysis of 7x0.5 tube in the longitudinal direction using 500x magnification. Upper: FWM heat T4684D6, tube by Euroflex, analysis by Admedes. Lower: FWM heat T4684D6, tube by Euroflex, analysis by MeKo

In addition, the maximum inclusion size and area fraction on the tube level was quantified by Admedes and MeKo. The results are listed in Table 5. Notably, the measured inclusions became significantly smaller than the Gen II specification limits for the mill product.

	Admedes		MeKo	
	Max. incl.	Area fraction	Max. incl.	Area fraction
Vascotube Heat T-4684D6.1	6 µm	0.68 %	5 µm	0.50 %
Euroflex Heat T-4684D6.1	7 µm	0.62 %	6 µm	1.00 %
Vascotube Heat T-3246D6	6 µm	0.66 %	4 µm	0.20 %
Euroflex Heat T-3246D6	6 µm	0.59 %	6 µm	0.55 %

Table 5: Max. inclusion size and area fraction of 7x0.5 tubes

Both Admedes and MeKo used a dogbone-shaped geometry (see Figure 6) to perform mechanical testing of the tubes according to ASTM F2516. Admedes tested heat treated samples while MeKo tested as cut samples. Both ran the tests at body temperature (37 °C). An example of the resulting tensile test diagrams can be seen in Figure 7.

Table 6 shows a summary of the mean values of each tube batch. The test results of each condition met the requirements.

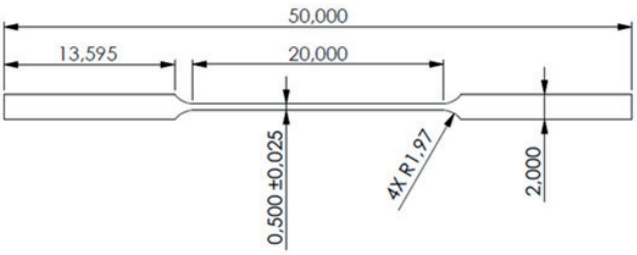
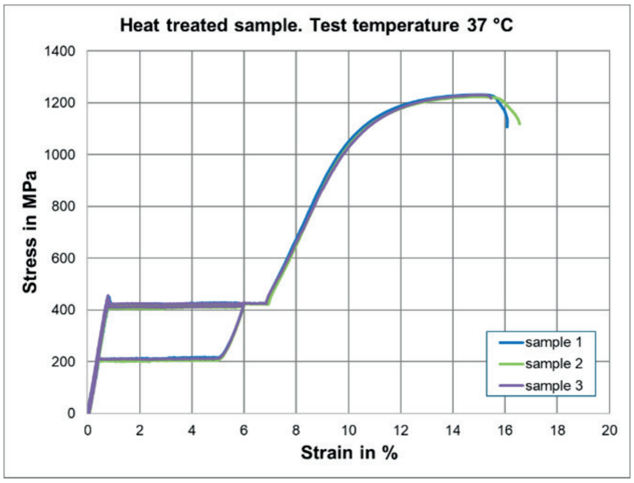


Fig. 6 : Dogbone shaped tensile test geometry



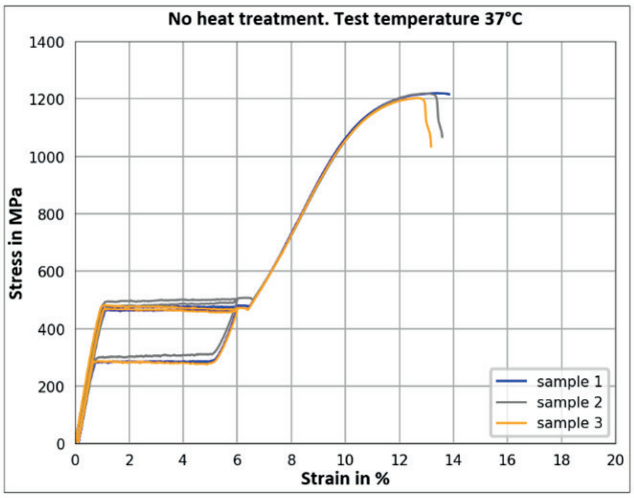


Fig. 7: Tensile test results. Upper : FWM heat T-3246D6, tube by Euroflex, test by Admedes. Lower: FWM heat T4684D6, tube by Euroflex, test by MeKo

Tested by	Tensile strength in MPa	UPS in MPa	LPS in MPa	Elf in %
Vascotube FWM heat T-4684D6				
Admedes (HT)	1248	393	182	16.5
MeKo	1180	509	335	18.1
Euroflex FWM heat T-4684D6				
Admedes (HT)	1280	401	189	14.1
MeKo	1214	448	290	13.6
Vascotube FWM heat T-3246D6				
Admedes (HT)	1162	410	203	19.3
MeKo	1200	496	344	17.7
Euroflex FWM heat T-3246D6				
Admedes (HT)	1228	422	209	16
MeKo	1190	486	291	15.4

Table 6: Mean mechanical properties

The A_f temperature was measured at tube manufacturers Euroflex and Vascotube using the so-called crush method based on ASTM F2082. A roughly 5 mm long tube section was cooled down from room temperature to -45°C in an alcohol bath, and crushed into an oval but not to exceed 3 % strain. The bath was then heated up to 20 °C with 5 °C/min. The A_f was extracted using the same tangent line method as described in ASTM F2082. The results met expectations and fulfilled the tube specification requirements.

	Admedes	MeKo
Vascotube Heat T-4684D6	-1.9 °C to 0.2 °C	-1.9 °C to 0.2 °C
Euroflex Heat T-4684D6	-9.0 °C to -4.0 °C	-8.6 °C to -3.1 °C
Vascotube Heat T-3246D6	-3.9 °C to -1.8 °C	-3.9 °C to -1.8 °C
Euroflex Heat T-3246D6	-0.3 °C to -1.8 °C	-1.1 °C to 2.0 °C

Table 7: Tube A_f temperature (crush method)

3 COMPONENT MANUFACTURING

Admedes and MeKo used the Gen II Nitinol tubing to evaluate the material in the component manufacturing process chain by producing the generic heart valve frame shown in Figure 8. Both manufacturers used their standard process chains including laser cutting, heat treatment, and surface removal processes.

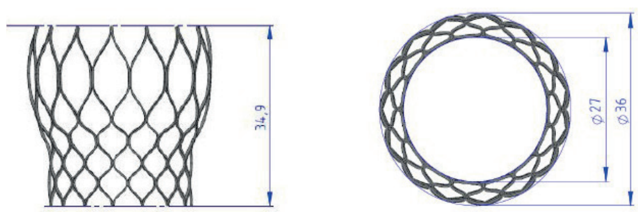


Fig 8: Genertic heart valve frame design in front and top view.

For each material lot MeKo manufactured 25 heart valve frames and Admedes manufactured between 15 and 22 frames. At both contract manufacturers no complications during laser cutting, heat treatment, or surface processes were observed.

3.1 Surface

The surface of the frames was inspected by light microscope and scanning electron microscope (SEM). Representative pictures of the outer diameter, inner diameter and tip ends are shown in Figure 9.

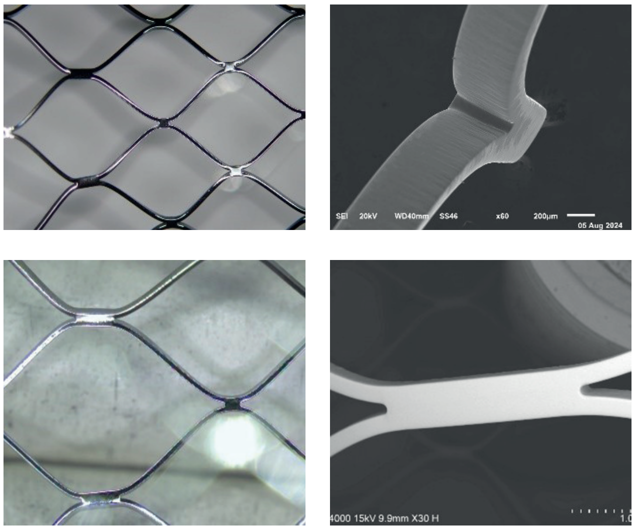


Fig. 9: Light microscope (left) and SEM (right).
Upper: Admedes frames. Lower: MeKo frames

The surface results met the expectations, as no material defects were detected.

3.2 Differential scanning calorimetry

DSC tests of the heat-treated components were conducted based on ASTM standard F2004. Each contract manufacturer used its standard temperature range. For Meko the range is between -80 °C and 60 °C and for Admedes between -180 °C and 100 °C.

The contract manufacturers chose expansion and heat treatment steps independently of each other. The DSC results of the devices are shown in Figure 10. The target A_f temperature of 20 °C ± 5 °C was achieved and the results fulfilled the expectations.

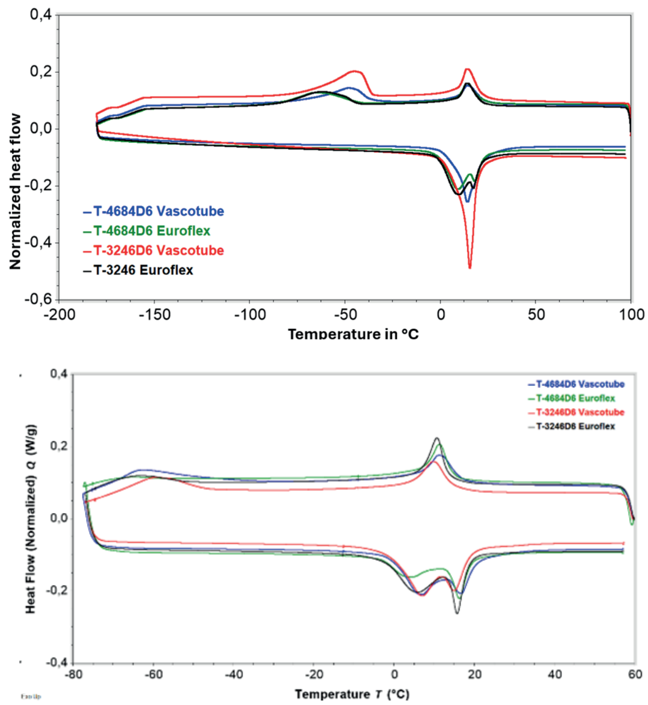
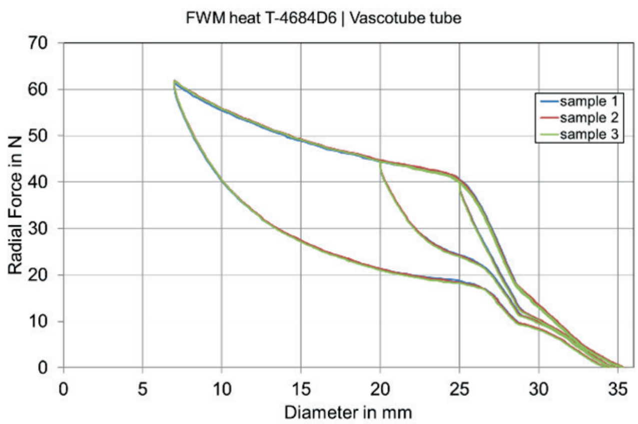


Fig. 10: DSC curves after heat treatment by Admedes (upper) and MeKo (lower)

3.3 Radial force

The heart valve frames with an outer diameter of 36 mm were crimped in three crimp cycles to a decreasing crimp diameter of 25 mm, 20 mm and 7 mm in order to determine the crimp behaviour and radial force. The results are shown in Figure 11. The test results of MeKo show a maximum diameter of 30 mm as it is the maximum diameter of the radial force tester. The results met the requirements.



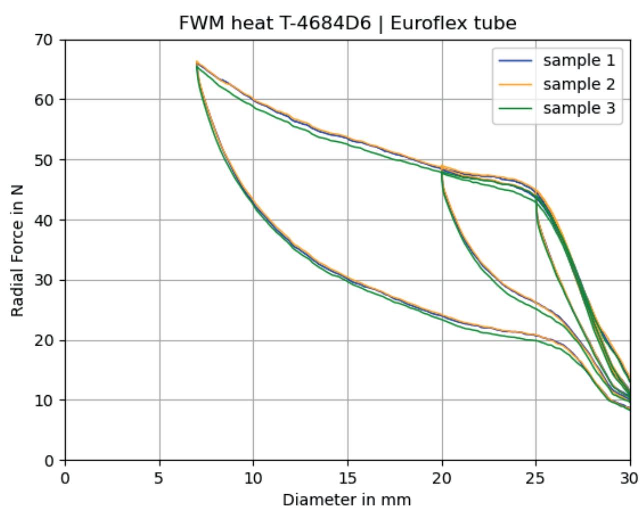


Fig.11: Radial force tests by Admedes (upper) and MeKo (lower)

3.4 Corrosion

The samples were subjected to corrosion testing using cyclic potentiodynamic polarization in phosphate-buffered saline solution at body temperature according to ASTM F2129. A saturated calomel electrode (SCE) was used as reference electrode.

Both Admedes and MeKo tested 5 samples from every material lot. All tests showed either high breakdown potentials over 900 mV or no breakdown (nB). Consequently, the parts can be considered corrosion resistant. Summarized results including mean rest potential E_r, minimum breakdown potential E_b and vertex potential E_v are listed in Table 8.

	E_r in mV (SCE)	E_b in mV (SCE)	E_v in mV
Admedes HVF | Vascotube | Heat T-4684D6.1	-435	nb	1000
Admedes HVF | Euroflex | Heat T-4684D6.1	-436	nb	1000
Admedes HVF | Vascotube | Heat T-3246D6	-424	nb	1000
Admedes HVF | Euroflex | Heat T-3246D6	-392	nb	1000
MeKo HVF | Vascotube | Heat T-4684D6.1	-424	963	1300
MeKo HVF | Euroflex | Heat T-4684D6.1	-414	986	1300
MeKo HVF | Vascotube | Heat T-3246D6	-409	971	1300
MeKo HVF | Euroflex | Heat T-3246D6	-423	1002	1300

Table 8: Corrosion test of heart valve frames

4 CONCLUSION

Within the context of the PRIME project Gen II Nitinol material was supplied by FWM. The objective was to verify whether the material functioned effectively in the processes of each company along the process chain and whether the resulting products meet the quality requirements. Multiple Nitinol Gen II tube sizes were manufactured. In this whitepaper tubes with outer diameter of 7 mm and wall thickness of 0.5 mm were investigated and processed to heart valve frames.

The material investigation began with the melt, which complied with the relevant standard ASTM F2063 and FWM internal specifications. The produced bar machinability allowed for gun drilling and subsequent fabrication into tubes. No complications were observed during tube drawing process by both tube suppliers Euroflex and Vascotube. The manufactured tubes met all specified mechanical and dimensional requirements. Furthermore, these tubes served as suitable semi-finished product for device manufacturing, with all analyzed results aligning with the expected quality and performance criteria. Specifically, the results of DSC, radial force and corrosion tests were inconspicuous, and the surface quality met all quality requirements.

Based on the results compiled in this paper, the supplied Nitinol bar from FWM met ASTM F2063 and Gen II requirements and can be used as a base material for medical devices. The addition of FWM as another ingot supplier also ensures a robust and independent ingot supply chain, offering clear benefits to tube manufacturers, medical device contract manufacturers, and their clients.

Looking ahead, the tube investigations will be extended to fatigue tests. In addition, other tube sizes processed to neurovascular stents and peripheral stents made from this material have also shown positive outcomes and the results are planned to be introduced in the near future.

5 ACKNOWLEDGMENTS

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6 REFERENCES

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