Assessment of component and tubing performance made of Fort-Wayne Metals Vacuum-Arc-Remelted Gen I Ingot for Tubing

by Admedes GmbH, Fort Wayne Metals, Euroflex GmbH, G.RAU GmbH & Co KG, MeKo Manufacturing e.K., Vascotube GmbH

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 (FWM) 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 the new Nitinol source for tube processability and device manufacturing requirements.

The project includes different melting methods resulting in different material grades as well as different tube sizes. This part focuses on so-called Gen I material produced from Vacuum-Arc-Remelted Nitinol.

Keywords: Nitinol, VAR, Tube, Component

1 VAR INGOT AND MILL PRODUCT

At Fort Wayne Metals (FWM) so-called Gen I material was produced using the vacuum-arc remelting (VAR) process. The VAR ingots (heat T-4041T16.6 and T-5111B16.1.2) used for the following investigations 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	Heat T-4041T16.6	Heat T-5111B16.1.2
Ni	54.5 to 57.0	56.0	56.1
C, max.	0.040	0.003	0.004
Co, max.	0.050	<0.003	<0.003
Cu, max.	0.010	0.005	0.002
Cr, max.	0.010	<0.003	<0.003
H, max.	0.005	<0.001	<0.001
Fe, max.	0.050	0.006	<0.002
Nb, max.	0.025	<0.003	<0.003
N, max.	0.005	0.004	0.001
O, max.	0.040	0.021	0.018

Table 1: Chemical composition requirements and analysis.

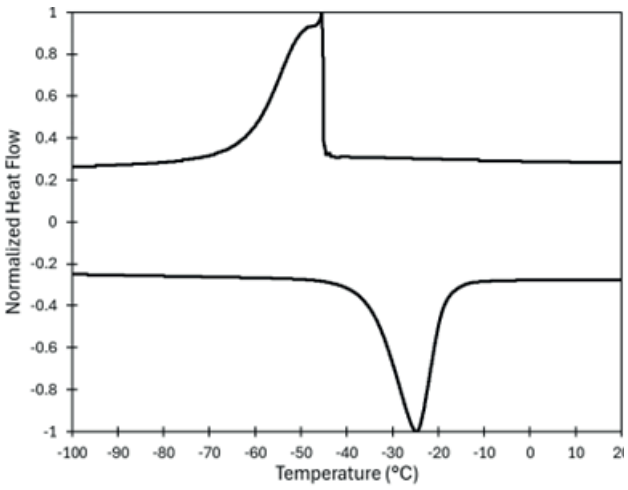


Fig. 1: Differential scanning calorimetry of fully annealed heat T-4041T16.6.

Figure 1 presents a differential scanning calorimetry (DSC) curve of heat T-4041T16.6 in fully annealed condition tested in accordance with ASTM F2004. The corresponding Austenitic start (As) transformation temperature was -35 °C and Austenitic finish (Af) temperature was -19 °C. The As transformation temperature range requirement of ASTM F2063 specifying a maximum range of 20 °C was fulfilled. In Table 2 transformation temperatures of both heats are listed.

Heat	As in °C	Af in °C
T-4041T16.6	-35	-19
T-5111B16.1.2	-19	-7

Table 2: Ingot As and Af temperatures.

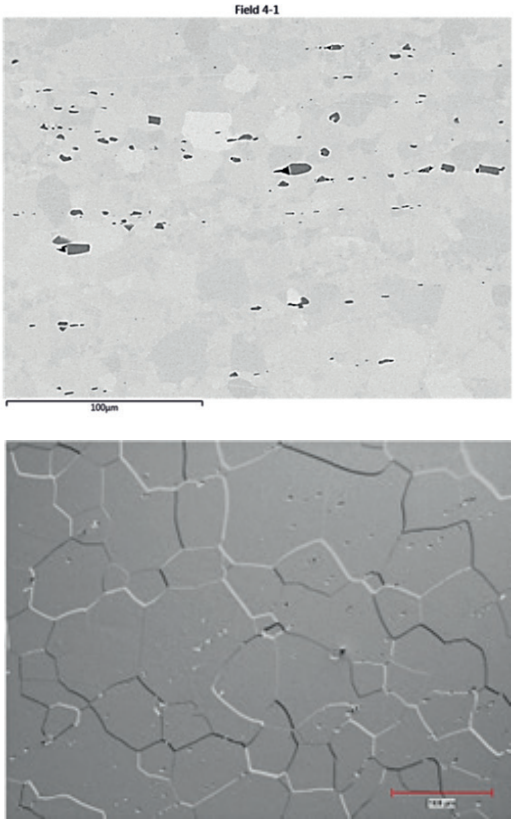


Fig. 2: Micrograph of mill product (T-4041T16.6) indicating inclusions (upper) and grains (lower).

Figure 2 shows representative micrographs in annealed condition of mill product of heat T-4041T16.6. The upper part indicates that the inclusion size satisfied the ASTM F2063 requirements. The maximum inclusion size and area fractions are summarized in Table 3. In addition, the ASTM grain size number of the mill product of both heats (T-4041T16.6, see Figure 2 lower, and T-5111B16.1.2) was 6 and fulfilled ASTM F2063 requirement of 4 or finer.

	ASTM F2063	T-4041T16.6	T-5111B16.1.2
Max. inclusion	39 µm	34.5 µm	37.7 µm
Max. area fraction	2.8 %	1.6 %	1.4 %

Table 3: Inclusion size and area fraction of VAR-EBR ingots.

2 TUBE MANUFACTURING

The tube size with outer diameter of 2.00 mm and wall thickness of 0.22 mm was manufactured by Euroflex and Vascotube according to standard tube specifications, see Table 4. Each manufactured tube batch was shipped to medical device contract manufacturers Admedes and MeKo. Both medical device contract manufacturers conducted incoming inspection by examining the microstructure and mechanical properties of each batch.

Tube sizes	2.00 mm x 0.22 mm
UTS min.	1100 MPa
UPS min. (at 3%)	380 MPa
Elongation at fracture min.	12 %
Max. residual elongation after 6 %	0.3 %
Af max.	15 °C

Table 4: Tube specifications. Mechanical properties tested according to ASTM F2516 at 22 °C.

Tube (mm)		Admedes	MeKo
2.00 x 0.22 Euroflex	Max. inclusion	10 µm	8 µm
	Area fraction	0.63 %	0.40 %
2.00 x 0.22 Vascotube	Max. inclusion	8 µm	4 µm
	Area fraction	0.64 %	0.20 %

Table 5: Max. inclusion size and area fraction of VAR tubing.

Figure 3 and Figure 4 show the microstructure of the finished tubing in longitudinal direction. The maximum inclusion size and area fraction are listed in Table 5.

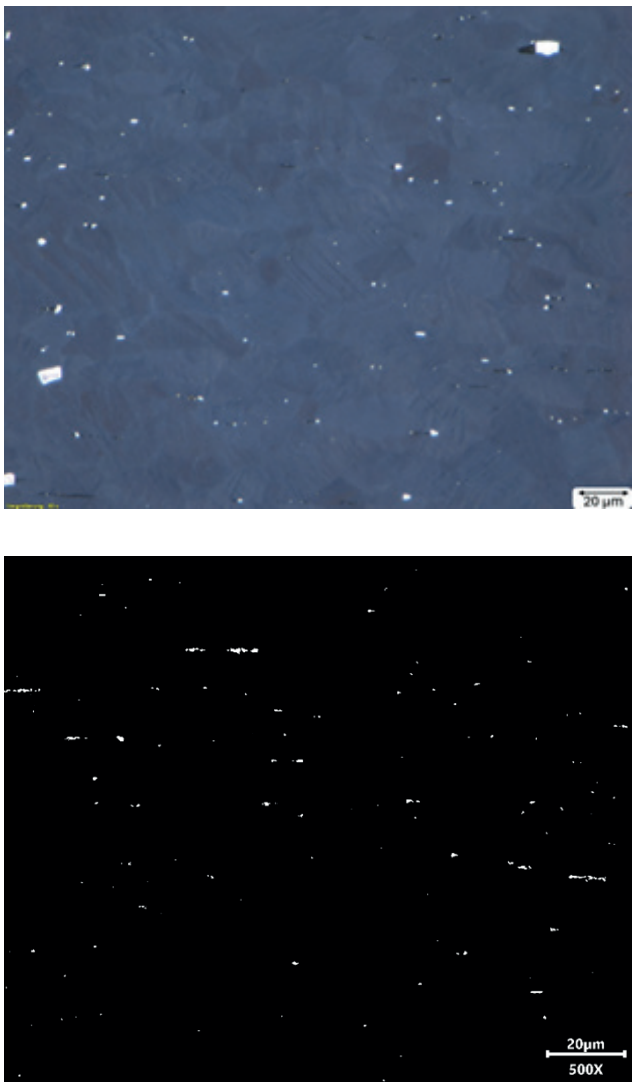


Fig. 3: Inclusion analysis of 2 mm x 0.22 mm tube by Euroflex in longitudinal direction using 500x magnification. Upper: Analysis by Admedes. Lower: Analysis by MeKo.

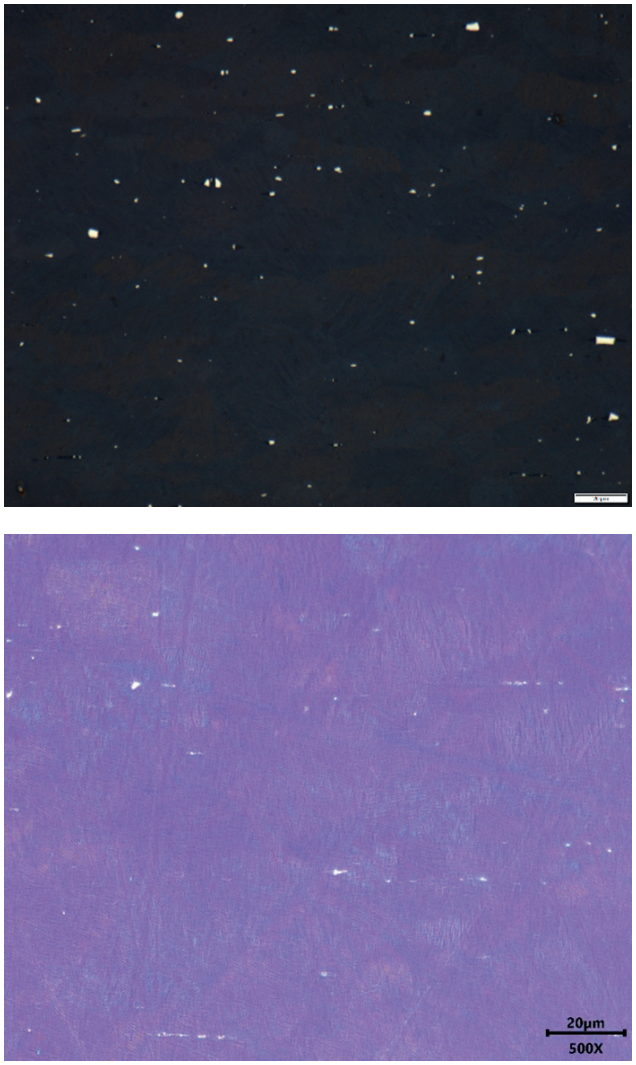


Fig. 4: Inclusion analysis of 2 mm x 0.22 mm tube by Vascotube in longitudinal direction using 500x magnification. Upper: Analysis by Admedes. Lower: Analysis by MeKo.

Admedes and MeKo used a dogbone-shaped geometry (see Figure 5) to perform mechanical testing of tubes according to ASTM F2516. Admedes ran the tests at room temperature (22 °C) and MeKo at body temperature (37 °C). Resulting tensile test diagrams are shown in Figure 6 and Figure 7. A summary of mean mechanical properties including results from Euroflex and Vascotube are listed in Table 6. In contrast to the dogbone sample geometry having a 0.5 mm thin strut width, Euroflex and Vascotube tested full circumference tube samples. Each company used its own test conditions. The differences observed align with the expectations.

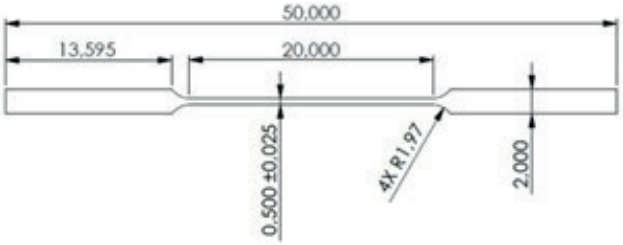
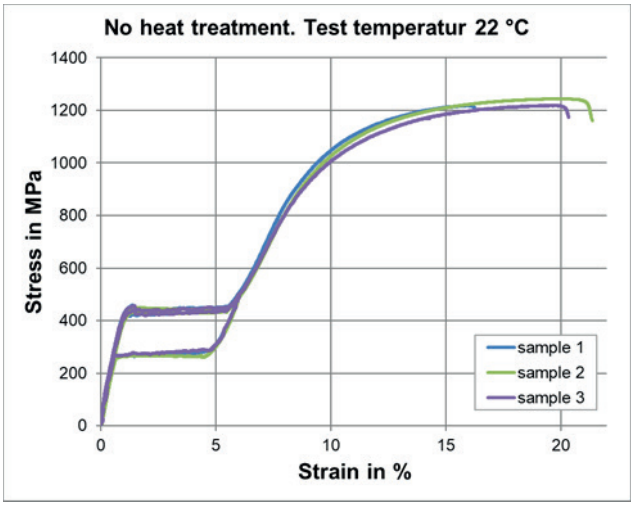


Fig. 5: Geometry of dogbone used for tensile testing.



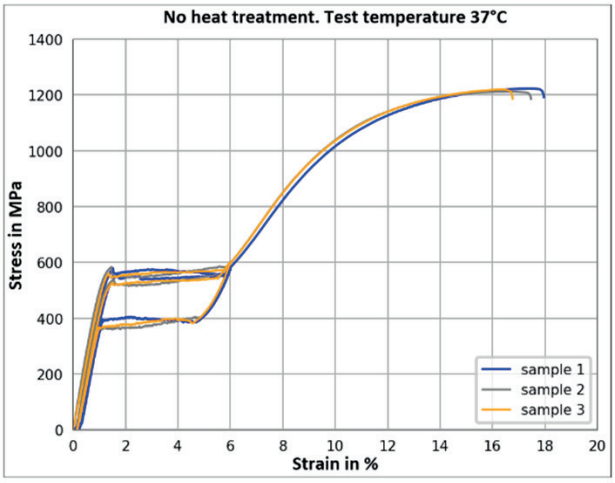
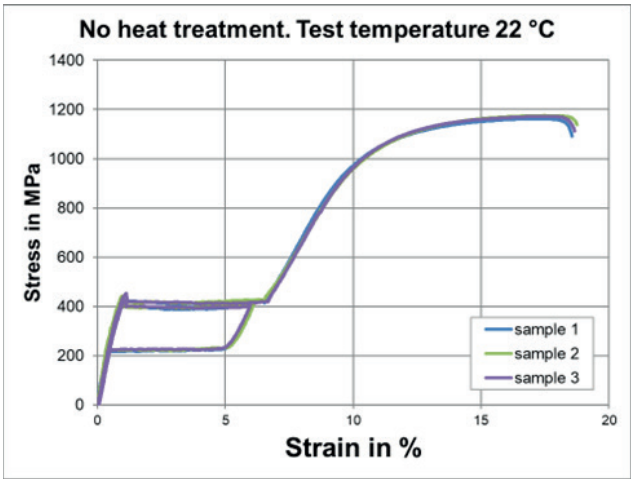


Fig. 6: Tensile test results of Euroflex tube. Upper: Admedes at 22 °C. Lower: MeKo at 37 °C.



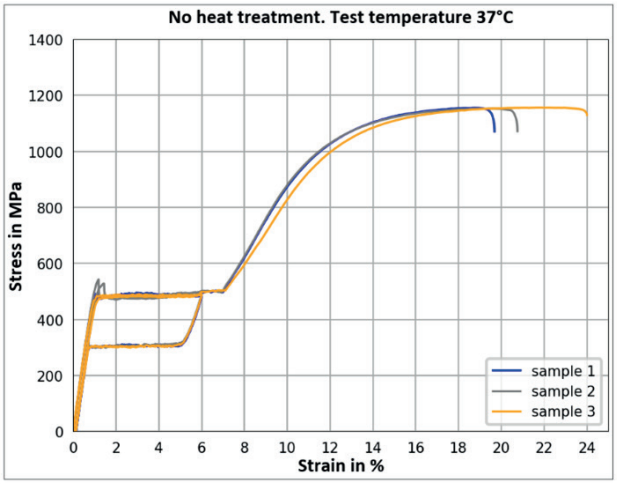


Fig. 7: Tensile test results of Vascotube tube. Upper: Admedes at 22 °C. Lower: MeKo at 37 °C.

The transformation temperature was measured using the so-called crush method. It is based on ASTM F2082. A tube section approximately 5 mm long was cooled down in an alcohol bath to -55 °C, the section was crushed into an oval but did not exceed 3 % strain. The bath was heated up to 20 °C with 5 °C/min. The Af was determined using the same tangent line method as described in ASTM F2082. The results are listed in Table 7 and are within the tube specification.

Mech. Properties	UTS in MPa	UPS in MPa	LPS in MPa	Ef in %
2.00 mm x 0.22 mm Euroflex tube				
Admedes 22 °C	1227	442	271	19.3
MeKo 37 °C	1216	570	397	17.5
Euroflex 22 °C	1168	452	237	27.5
2.00 mm x 0.22 mm Vascotube tube				
Admedes 22 °C	1170	413	223	18.7
MeKo 37 °C	1155	489	306	21.5
Euroflex 22 °C	1203	469	223	20.7

Table 6: Average tube mechanical properties.

	Euroflex	Vascotube
2.00 mm x 0.22 mm	-17.1 °C	-6.4 °C

Table 7: Average Af temperature of tubes measured by tube suppliers.

3 COMPONENT MANUFACTURING

Admedes and MeKo used the Gen I Nitinol tubing to evaluate the material in the stent manufacturing process chain by producing the generic stent design shown in Figure 8. Both manufacturers used their standard process chains including laser cutting, heat treatment and removing surface processes. The product at the end of all processes is an electropolished stent with an inner diameter of 8 mm.

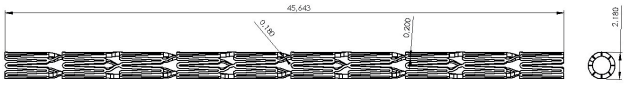


Fig. 8: Unexpanded peripheral stent.

In total, MeKo and Admedes manufactured 87 peripheral stents. At both contract manufacturers there were no complications during laser cutting, heat treatment, or surface processes.

3.1 SURFACE

The surface of the stents was inspected by light microscope and scanning electron microscope (SEM).

Representative pictures of the stent outer diameter, inner diameter and tip ends are shown in Figure 9 and Figure 10. In summary, there were no material related defects observed at either contract manufacturer.

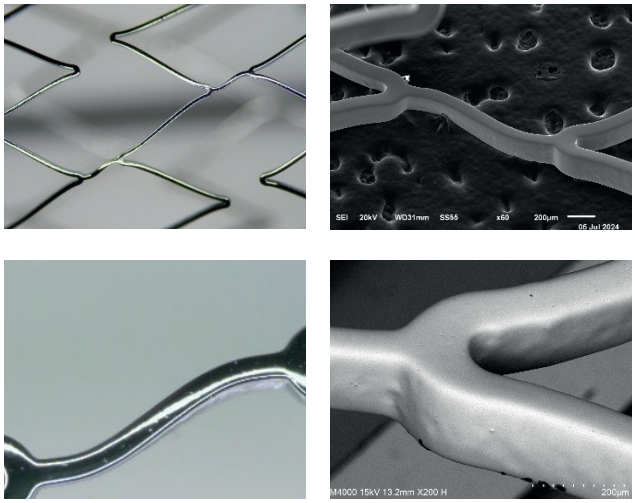


Fig. 9: Light microscope (left) and SEM (right) of peripheral stents made from Euroflex tube. Upper: Admedes. Lower: MeKo.

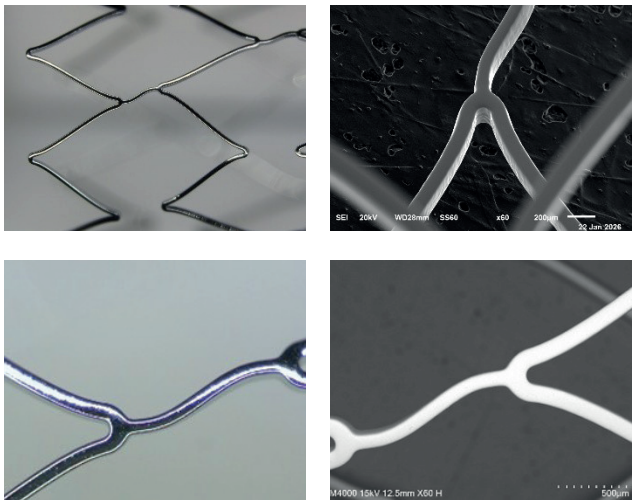


Fig.10: Light microscope (left) and SEM (right) of peripheral stents made from Vascotube tube. Upper: Admedes. Lower: MeKo.

3.2 DIFFERENTIAL SCANNING CALORIMETRY

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

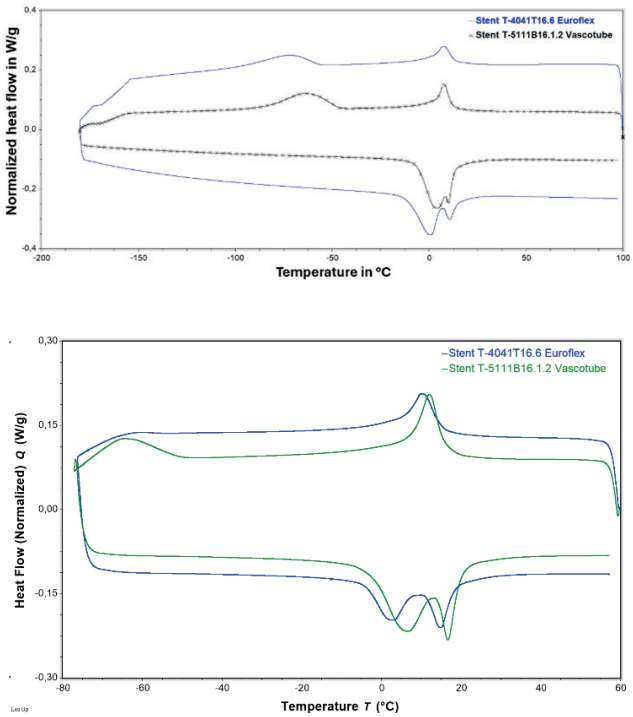


Fig. 11: DSC curves of stents by Admedes (upper) and MeKo (lower).

The contract manufacturers chose expansion and heat treatment steps independently of each other. The DSC results are shown in Figure 11 and fulfilled expectations.

3.3 RADIAL FORCE

The peripheral stents were crimped to a diameter of 4 mm with three crimping repetitions, see Figure 12 and Figure 13. Due to the differences in Af temperature and strut width, the radial force results align with the anticipated outcomes.

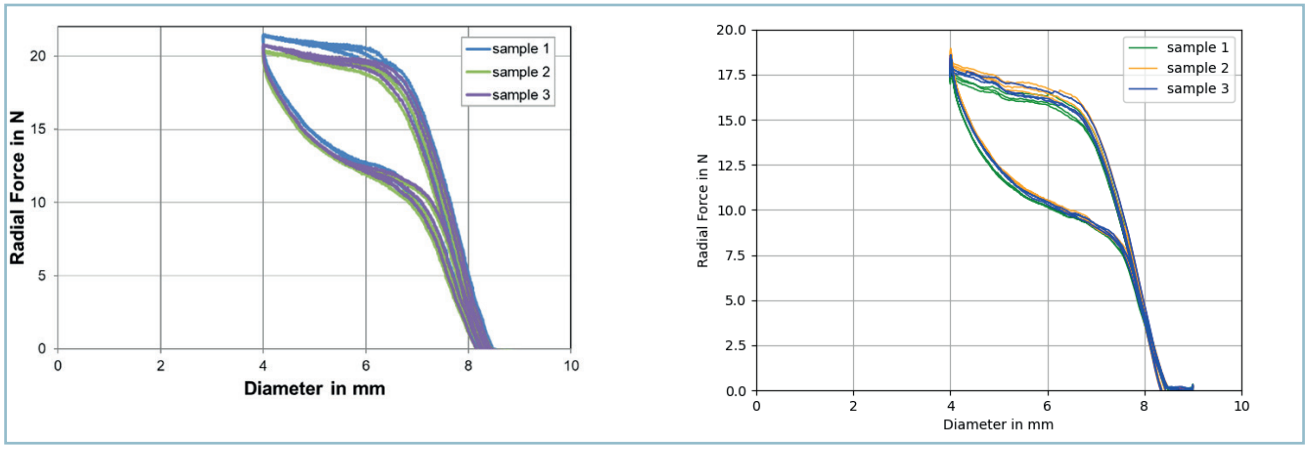


Fig. 12: Radial force tests of peripheral stents from Euroflex tube by Admedes (left) and MeKo (right).

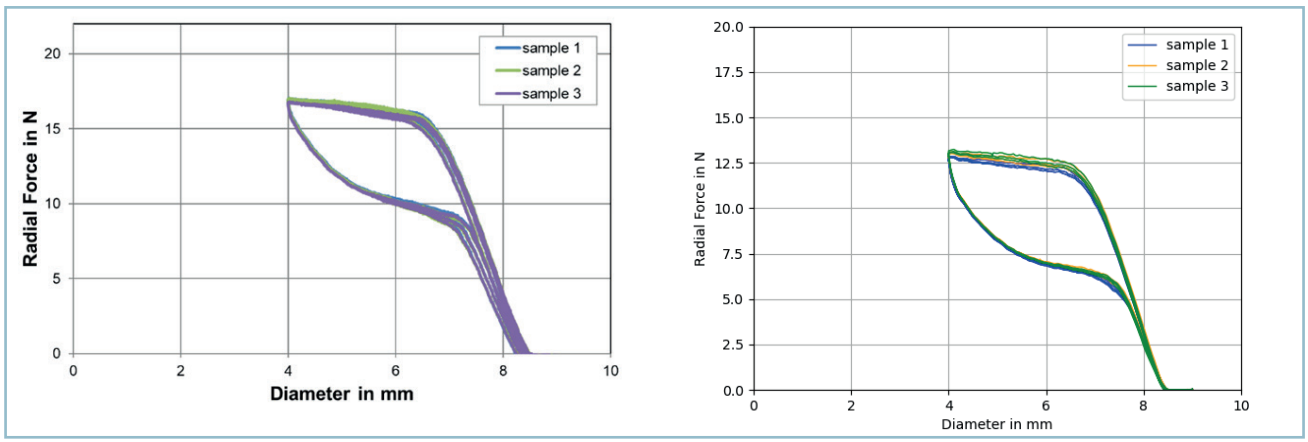


Fig. 13: Radial force tests of peripheral stents from Vascotube tube by Admedes (left) and MeKo (right).

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 the reference electrode. Admedes and MeKo tested five stent samples from every tube lot. The corrosion test results align with the aniticipated outcomes.

No.	Sample	Er in mV (SCE)	Eb in mV (SCE)	Ev in mV
1	Admedes stent	-167	nB	1000
2	Admedes stent	-187	nB	1000
3	Admedes stent	-353	nB	1000
4	Admedes stent	-371	nB	1000
5	Admedes stent	-354	nB	1000
1	MeKo stent	-433	1005	1300
2	MeKo stent	-437	995	1300
3	MeKo stent	-446	1000	1300
4	MeKo stent	-430	999	1300
5	MeKo stent	-323	1031	1300

Table 8: Corrosion test results of peripheral stents of Euroflex tube.

The results of peripheral stents are summarized in Table 8 and Table 9. There were either high breakdown potentials over 900 mV or no breakdown (nB) observed, meaning the parts were considered corrosion resistant.

No.	Sample	Er in mV (SCE)	Eb in mV (SCE)	Ev in mV
1	Admedes stent	-506	nB	1000
2	Admedes stent	-280	nB	1000
3	Admedes stent	-477	nB	1000
4	Admedes stent	-486	nB	1000
5	Admedes stent	-244	nB	1000
1	MeKo stent	-465	952	1300
2	MeKo stent	-462	983	1300
3	MeKo stent	-464	1005	1300
4	MeKo stent	-462	994	1300
5	MeKo stent	-468	942	1300

Table 9: Corrosion test results of peripheral stents of Vascotube tube.

4 CONCLUSION

Within the context of the PRIME project Gen I Nitinol was melted with VAR method by Fort Wayne Metals. The objective was to verify whether the material functions effectively in the processes of each company along the process chain and whether the resulting products meet the quality requirements. In this white paper tubes with outer diameter of 2 mm and wall thickness of 0.22 mm of two melts were manufactured by Euroflex and Vascotube and further processed to stents by Admedes and MeKo.

The material investigation began with the melt, which complied with the relevant standard ASTM F2063. The produced bar machinability allowed gun drilling and subsequent fabrication into tubes. No complications were observed during tube drawing process by Euroflex or Vascotube. The manufactured tubes met all specified mechanical and dimensional requirements. Furthermore, these tubes have been determined to serve as suitable semi-finished products for device manufacturing, with all analyzed results aligning with the expected quality and performance criteria. In particular, 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 Fort Wayne Metals VAR material which met ASTM F2063 requirements, is deemed suitable as a base material for manufacturing medical devices. The addition of Fort Wayne Metals as another ingot supplier for VAR material also ensures a robust and independent ingot supply chain, offering clear benefits to tube manufacturers, medical device contract manufacturers and their clients.

5 ACKNOWLEDGMENTS

The investigations discussed in this paper were funded by the partners Fort Wayne Metals Research Products, Vascotube a Cirtec Company, Euroflex GmbH, G.RAU GmbH & Co KG, Admedes GmbH and MeKo Manufacturing e.K..

6 REFERENCES

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