Working Report 2008-73

Manufacturing of the Canister Shells T54 & T55

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October 2008

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ABSTRACT

This report constitutes a summary of the manufacturing test of the disposal canister copper shells T54 and T55.

The copper billets were manufactured at Luvata Pori Oy, Finland. The hot-forming and machining of the copper shells were made at Vallourec & Mannesmann Tubes, Reisholz mill, Germany. The shells were manufactured with the pierce and draw method. Both of the pipes were manufactured separately in two phases. The first phase consisted of following steps: preheating of the billet, upsetting, piercing and the first draw with mandrel through drawing ring. After cooling down the block is measured and machined in case of excessive eccentricity or surface defects. In the second phase the block is heated up again and expanded and drawn in 6 sequences. In this process the pipe inside dimension is expanded and the length is increased in each step. Before the last, the 6th step, the bottom of the pipe is deformed in a sequence of special processes.

During the manufacture of the first pipe, T54, some difficulties were detected with the centralization of the billet before upsetting. For the second manufacture of the T55, an additional steering ring was made and the result was remarkably more coaxial.

After the manufacture and non-destructive inspections the shells were cut in pieces and three parts of each shell were taken for destructive testing. The three inspected parts were the bottom plate, a ring from the middle of the cylinder and a ring from the top of the cylinder. The destructive testing was made by Luvata Pori Oy.

In spite of some practical difficulties and accidents during the manufacturing process, the results of the examinations showed that both of the test produced copper shells fulfilled all the specified requirements as for soundness (integrity), mechanical properties, chemical composition, dimensions, hardness and grain size.

Keywords: pierce & draw -method, copper canister, disposal of spent nuclear fuel

KAPSELIVAIPPOJEN T54 & T55 VALMISTUS

TIIVISTELMÄ

Tämä raportti antaa yhteenvedon loppusijoituskapselin kuparivaippojen T54 ja T55 valmistuskokeesta.

Valmistuskokeessa käytetyt kupariaihiot valmisti Suomessa Luvata Pori Oy. Kuparivaippojen kuumamuokkauksen ja koneistukset suoritti Vallourec & Mannesmann Tubes -yhtiön putkitehdas Reisholzissa, Saksassa. Valmistusmenetelmänä oli ns. pisto ja veto -tekniikka. Molemmat putket valmistettiin erikseen kahdessa vaiheessa. Ensimmäinen vaihe koostui seuraavista askelista: valanteen esikuumennus, tyssäys, pisto sekä ensimmäinen vetovaihe tuurnalla vetorenkaan läpi. Jäähdytyksen jälkeen kappale mitataan ja, mikäli se on liian epäkeskeinen tai pinnassa on vikoja, koneistetaan sorvissa. Toisessa vaiheessa kappale kuumennetaan uudelleen ja sille suoritetaan kuudessa vaiheessa sisähalkaisijan laajennus tuurnalla ja veto vetorenkaan lävitse. Tässä prosessissa sisähalkaisija kasvatetaan ja putken pituus kasvaa kussakin askeleessa. Ennen viimeistä, kuudetta vetoaskelta, putken pohja muokataan sarjalla erikoisprosesseja.

Ensimmäisen putken, T54, valmistuksen yhteydessä todettiin vaikeuksia valanteen keskittämisessä ennen tyssäystä. Toisen valmistuskokeen, T55, suoritusta varten tyssäysastiaan tehtiin erityinen keskitysohjain, ja lävistys tapahtui merkittävästi paremmin keskitettynä.

Valmistusvaiheiden ja ainetta rikkomattomien tarkastusten jälkeen kuparivaipat katkottiin osiin ja kolme näytepalaa kummastakin lieriöstä otettiin rikkoviin tutkimuksiin. Nämä osat olivat pohjapääty sekä keskivaiheilta ja yläpäästä otetut näyterenkaat. Ainetta rikkovat tutkimukset suoritti Luvata Pori Oy.

Tarkastusten tulokset osoittivat, että kuparivaipat täyttivät niille spesifioidut valmistusvaatimukset materiaalin eheyden, mekaanisten ominaisuuksien, kemiallisen koostumuksen, mittojen, kovuuden ja raekoon suhteen.

Avainsanat: pisto ja veto -menetelmä, kuparikapseli, käytetyn ydinpolttoaineen loppusijoitus

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1 INTRODUCTION

The technique to manufacture copper tubes for the KBS-3 concept of disposal of nuclear waste have been tested and developed since 1970th by SKB. Three techniques have been used; extrusion, forging and pierce & draw methods. All together more than 40 copper tubes have been manufactured with these methods.

SKB and Posiva have agreed on co-operation of manufacturing copper tubes with integrated bottom by pierce and draw method to determine manufacturing process in technical perspective and on the other hand to manufacture a tube/canister that fulfils safety criteria and metallurgical quality that is set for the canister. Main character is to have homogeneous copper canister with coexistent grain size in canister shell and bottom. Hot-forming process has been optimised and changed to reach optimal process for serial production

The aim with the project is to get more knowledge by controlling temperatures and other variable factors during pierce & draw process and test serial process. The project is a part of the SKB-Posiva Joint work programme on Canister and Encapsulation Technology. Clients are Canister Manufacturing at SKB and Development unit at Posiva.

This report constitutes a summary of the manufacturing tests of the disposal canister copper shells T54 and T55.

The copper billets were manufactured at Luvata Pori Oy, Finland. The quality plan for the billet casting in shown in App. 1 and the material certificates for each of the billets are shown in Apps. 2 and 3.

The hot-forming and machining of the copper shells were made at Vallourec & Mannesmann Tubes, Reisholz mill, Germany. The shells were manufactured with the pierce and draw method. After the manufacture and non-destructive inspections the shells were cut in pieces and three parts of each shell were taken for destructive testing. The parts were the bottom plate, a ring from the middle of the cylinder and a ring from the top of the cylinder. The destructive testing was made by Luvata Pori Oy.

The cylindrical parts of the remaining cylinders (totally 4 ex.) were delivered first to Canister Laboratory in Oskarshamn for NDT-testing and then to Posiva to be used as material for welding tests.

The results of the examinations showed that both of the test produced copper shells fulfilled all the specified requirements as for soundness (integrity), mechanical properties, chemical composition, dimensions, hardness and grain size. The applied specification was KTS 002 rev. 1, and procedures KT 0701 rev. 4, KT 0702 rev. 5, KT 0703 rev. 4, KT 0704 rev. 5, KT 0705 rev. 5, KT 0801 rev. 3, KT 1102 rev. 3 and KT 1103 rev. 3. The applied quality plan No. 1411 was applied. The quality plan for the pierce & draw process is attached as App. 4.

The reporting of the hot deformation, machining and non-destructive controls is compiled by Vallourec & Mannesmann Tubes. The Vallourec & Mannesmann controls reporting in made by Helmut Backes. The manufacture follow-up and metallurgical testing and reporting is made by Timo Välimäki, Jouko Lammi and Tuomas Renfors of Luvata Pori Oy and shown in Apps. 5, 6 and 7.

This summary report is compiled by Heikki Raiko of VTT from the written documentation produced by the organisations mentioned above and identified in the Appendices.

2 COPPER BILLETS

The billets were made by Luvata Pori Oy during spring 2007.

The billet for T54 has the cast identification 07-74-2-1. The length of the billet was as cast 3 070 mm and weight 15 278 kg. After taking the material test samples from top and bottom end, the both ends and the cylindrical surface were machined. The final dimensions after machining were; length 2 828 mm, diameter 827/830 mm and weight 13 640 kg.

Accordingly, the billet information for the T55 manufacture is as follows. The cast identifier was 07-75-1-1. The length of the billet was as cast 3 110 mm and weight 15 460 kg. After taking the material test samples from top and bottom end, the both ends and the cylindrical surface were machined. The final dimensions after machining were; length 2 792 mm, diameter 830/834 mm and weight 13 540 kg.

According to visual inspection the cylindrical surface had no indications. After machining, the both ends were dye penetrant tested. Top end had no indications but bottom end had one central indication after machining in both billets. The manufacturer's certificates for the both billets are attached to this report as Apps. 2 and 3. The certificates include photographs from the bottom end of the both billets after a sequence of machining. The billets are shown in Fig.1 below.

Figure 1. *The billets for T54 and T55 ready for transportation to hot forming at Luvata Pori Oy.*

3 HOT DEFORMATION OF THE CANISTER SHELLS

The billet sizes for T54 and T55 were: Do 827 mm and 830 mm and the length L 2828 mm and 2792 mm, respectively. The fabrication steps are as follows; first (T54 on 4.6.2007) the billet is upset into a D1135 mm die, and then the billet is pierced with a 690 mm diameter mandrel. Then the billet was drawn with a horizontal mandrel through a drawing ring. Now the billet was cooled down and dimensions were measured. Then, one month later, on 2.7.2007, a sequence of heating, expanding with wider mandrel and drawing was planned to be repeated for additional 5 times. In addition, between the last and the second last drawing the bottom was deformed in a few steps with special tools.

The same procedure was used to manufacture the T55. The first manufacturing phase was done on 12.11.2007 and the second on 10.12.2007. The billet for T55 was falling away from the gripping device of the crane before upsetting. However, this did not cause any damage.

3.1 Upsetting

The heating of the billet T54 was made in the furnace, whose temperature was 750 C. Then the billet was put into the pre-heated die. Upsetting was made in one phase and with one stroke. The die diameter was 1135 mm. The capacity of the press used for upsetting was used at the maximum limit. The phases of upsetting and piercing are shown schematically in Figure 2 and in reality in Figure 3. The die and pin are pre-heated by the previous manufacturing program of other pipes.

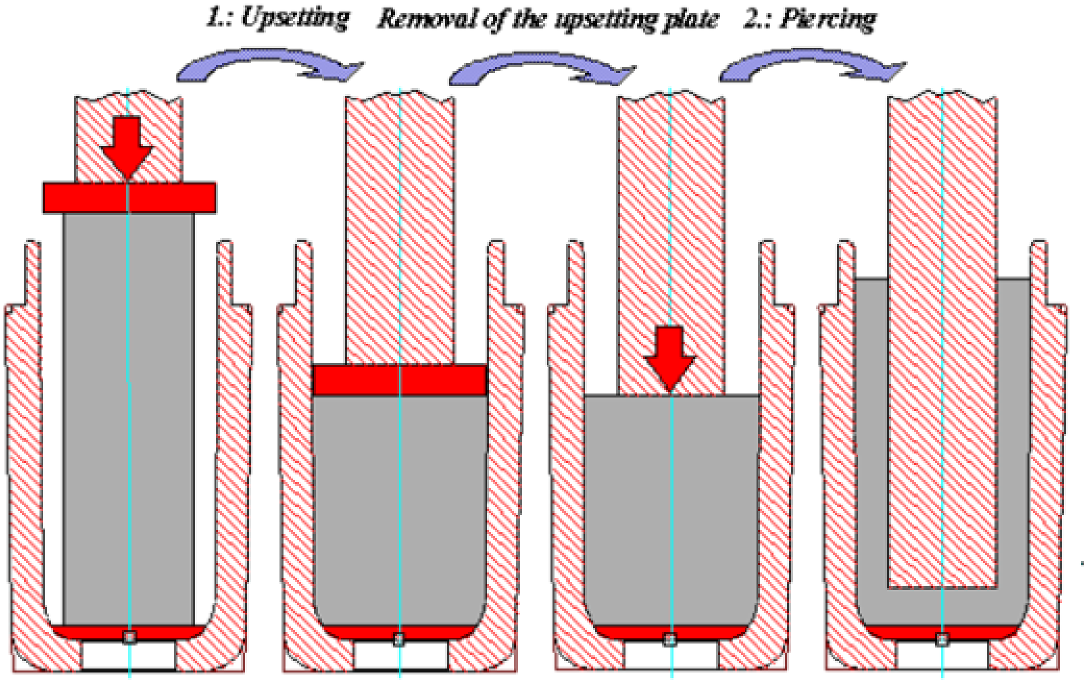


***Figure 2.** Phases of upsetting and piercing (Vallourec & Mannesmann Pipes).*

Out of the pre-heating furnace Into the die for upsetting

Removal of the centralising device Positioning of the upsetting plate

After upsetting And after piercing

Figure 3. *Photographs of upsetting and piercing the big copper billet T54 (Vallourec & Mannesmann Tubes).*

Later it was detected that the positioning of the billet of the T54 was somehow tilted or eccentric in the die and the billet had not filled the die perfectly after upsetting. That is why a new centralising device was made for the manufacture of the T55. The earlier and the new versions of the centralising device are shown in Figure 4.

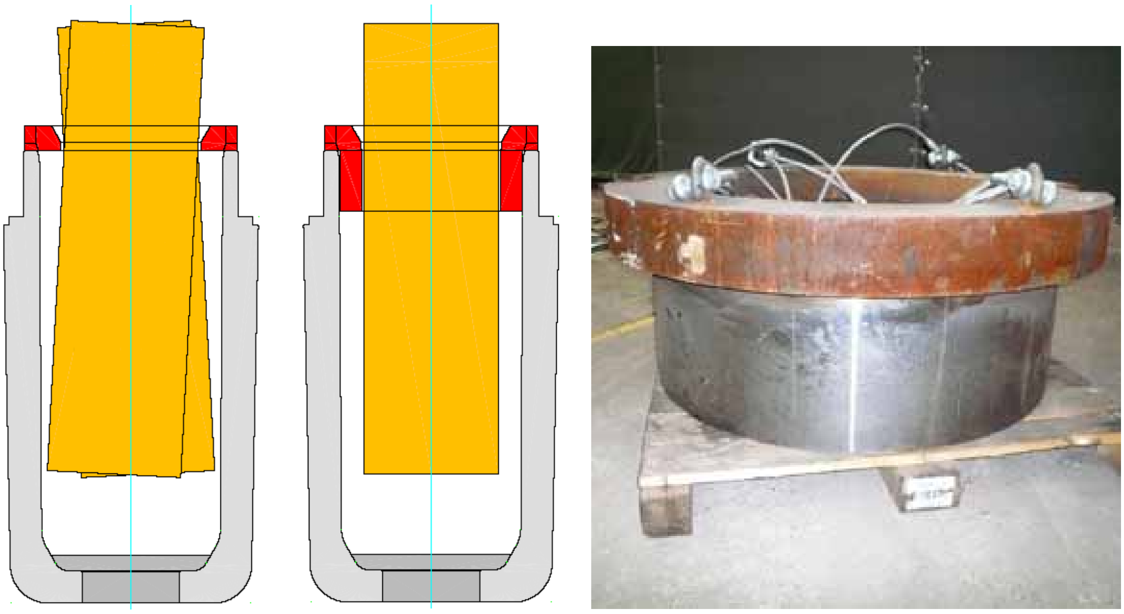


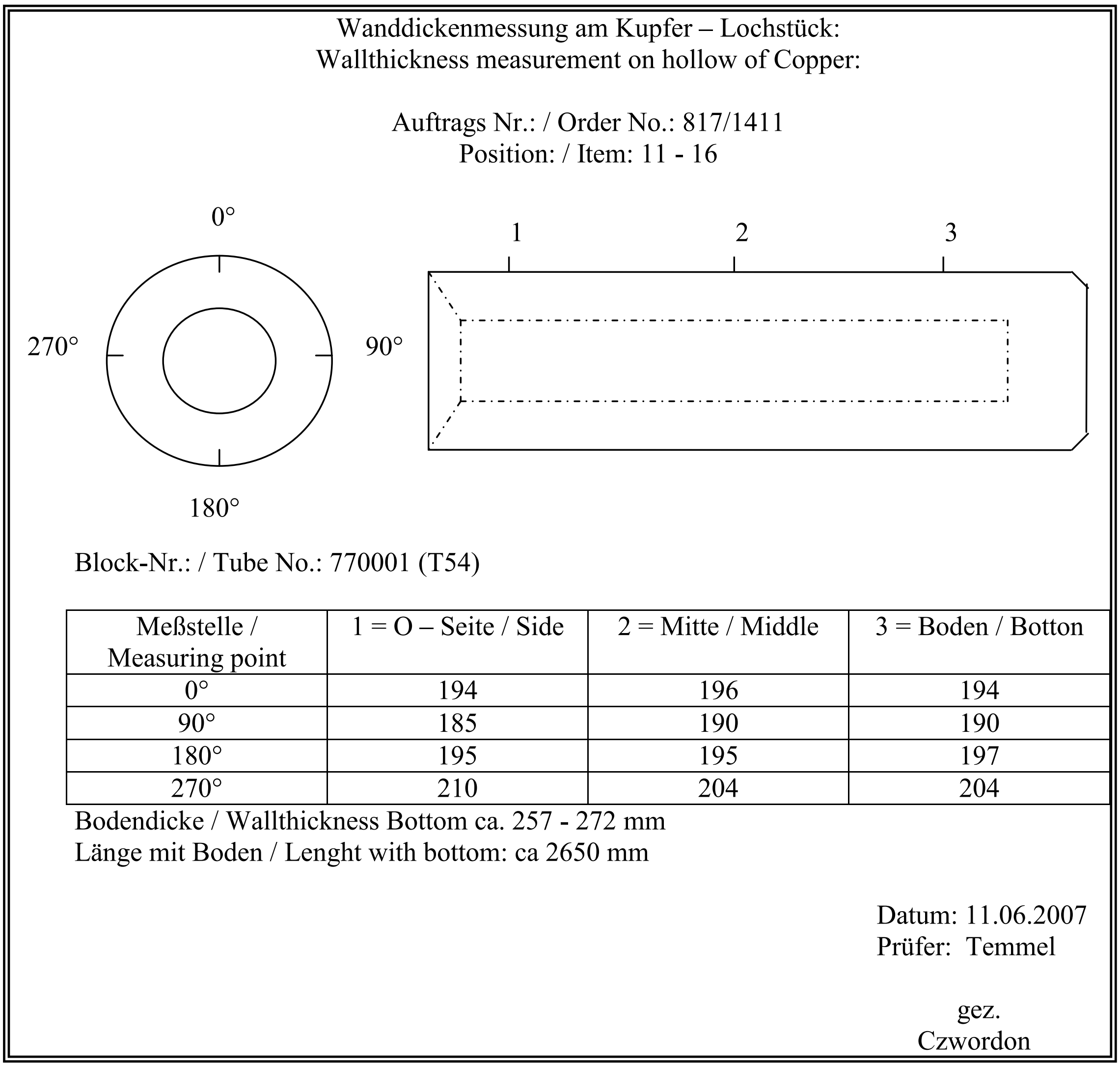
Figure 4. *The shorter centralising ring used for T54, left, and the new modified centralising ring used for T55, in the middle, and the new ring in the reality, on the right.*

The heating of the billet T54 was made in the furnace, whose temperature was 780 C. This was a planned change, because the force needed for upsetting the T54 was at the limit of the press. However, due to problems with the gripping device of the crane, there were some 10 minutes of delay and thus the billet temperature was practically the same as for the T54 during upsetting. The upsetting process went through without problems.

3.2 Piercing

Directly, after upsetting the billets were pierced. The upsetting plate on top of billet was removed and billet was pierced with D690 mm piercing mandrel. The phases of piercing are shown schematically in Figure 2 and in reality in Figure 3. The bottom thickness was adjusted to be about 260-270 mm. The upsetting, the piercing and the first pierce and draw step was made continuously in one phase without re-heating.

After the first pierce and draw step the billets were cooled down and the dimensions were controlled. The dimensions of T54 are given in Figure 5. The eccentricity of the pierced hollow was remarkably high; variation of wall thickness was from 25 mm, at maximum. This is why machining was made for the T54 ingot before following drawing phases. After machining the maximum variation of the wall thickness was 5 mm.



Wanddickenmessung am Kupfer – Lochstück:
Wallthickness measurement on hollow of Copper:

Auftrags Nr.: / Order No.: 817/1411
Position: / Item: 11 - 16

Block-Nr.: / Tube No.: 770001 (T54)

Meßstelle / Measuring point	1 = O – Seite / Side	2 = Mitte / Middle	3 = Boden / Botton
0°	194	196	194
90°	185	190	190
180°	195	195	197
270°	210	204	204

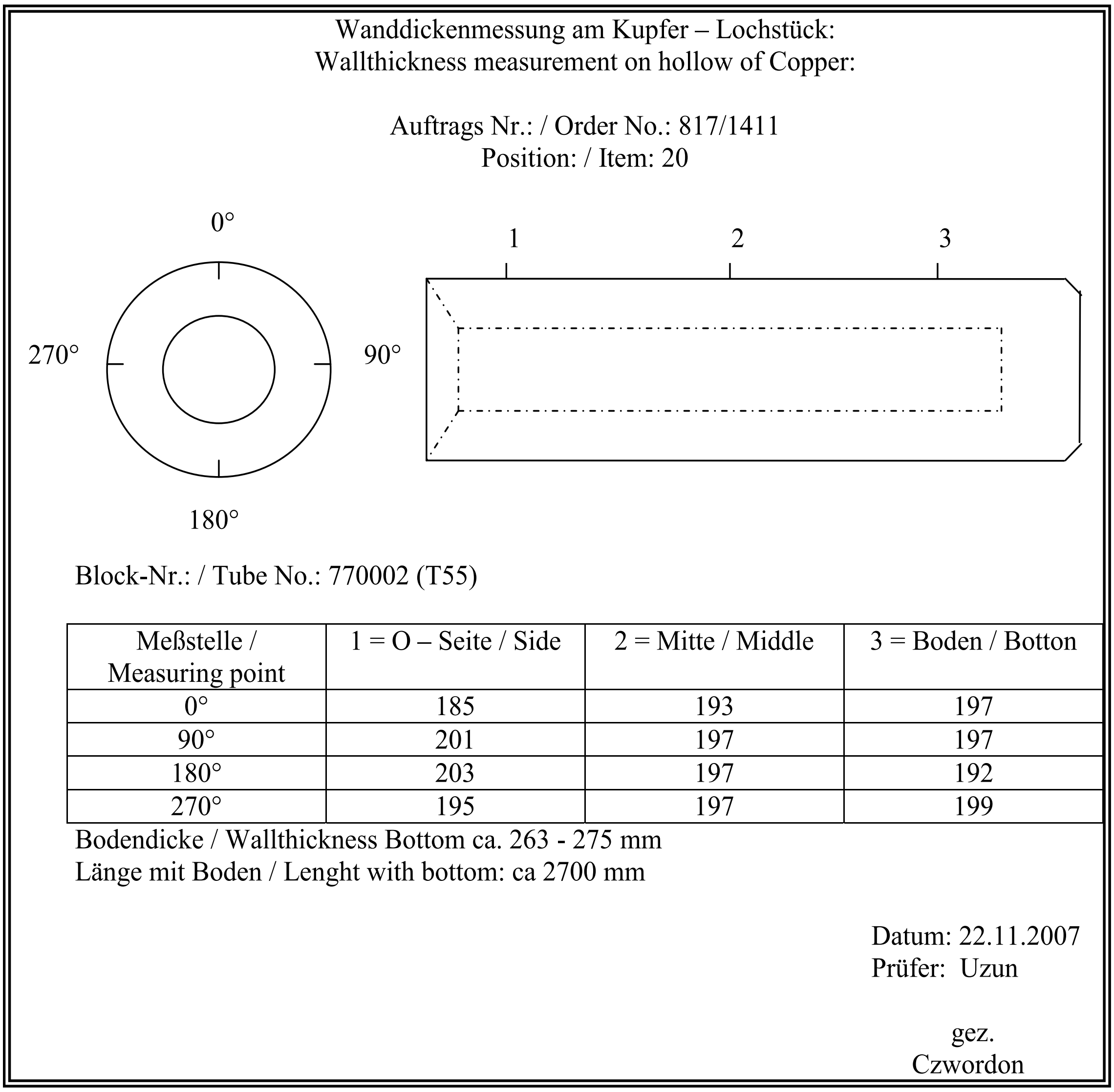
Bodendicke / Wallthickness Bottom ca. 257 - 272 mm
Länge mit Boden / Lenght with bottom: ca 2650 mm

Datum: 11.06.2007
Prüfer: Temmel

gez.
Czwordon

Figure 5. *The results of the dimensional control of the billet T54 after piercing.*

The upsetting of the T55 billet was made with better centralising and after that the piercing was also resulting to a better result. The results of the dimensional test of the T55 are shown in Figure 6.



Wanddickenmessung am Kupfer – Lochstück:
Wallthickness measurement on hollow of Copper:

Auftrags Nr.: / Order No.: 817/1411
Position: / Item: 20

Block-Nr.: / Tube No.: 770002 (T55)

Meßstelle / Measuring point	1 = O – Seite / Side	2 = Mitte / Middle	3 = Boden / Botton
0°	185	193	197
90°	201	197	197
180°	203	197	192
270°	195	197	199

Bodendicke / Wallthickness Bottom ca. 263 - 275 mm
Länge mit Boden / Lenght with bottom: ca 2700 mm

Datum: 22.11.2007
Prüfer: Uzun

gez.
Czwordon

Figure 6. *The results of the dimensional control of the billet T55 after piercing.*

T55 was also machined after the first manufacturing phase to get a better centralising. In addition, in the top part of the T55 cylinder there were some material faults that should be removed by machining. For details, see Figure 7. After machining the maximum variation of the wall thickness was 5 mm.

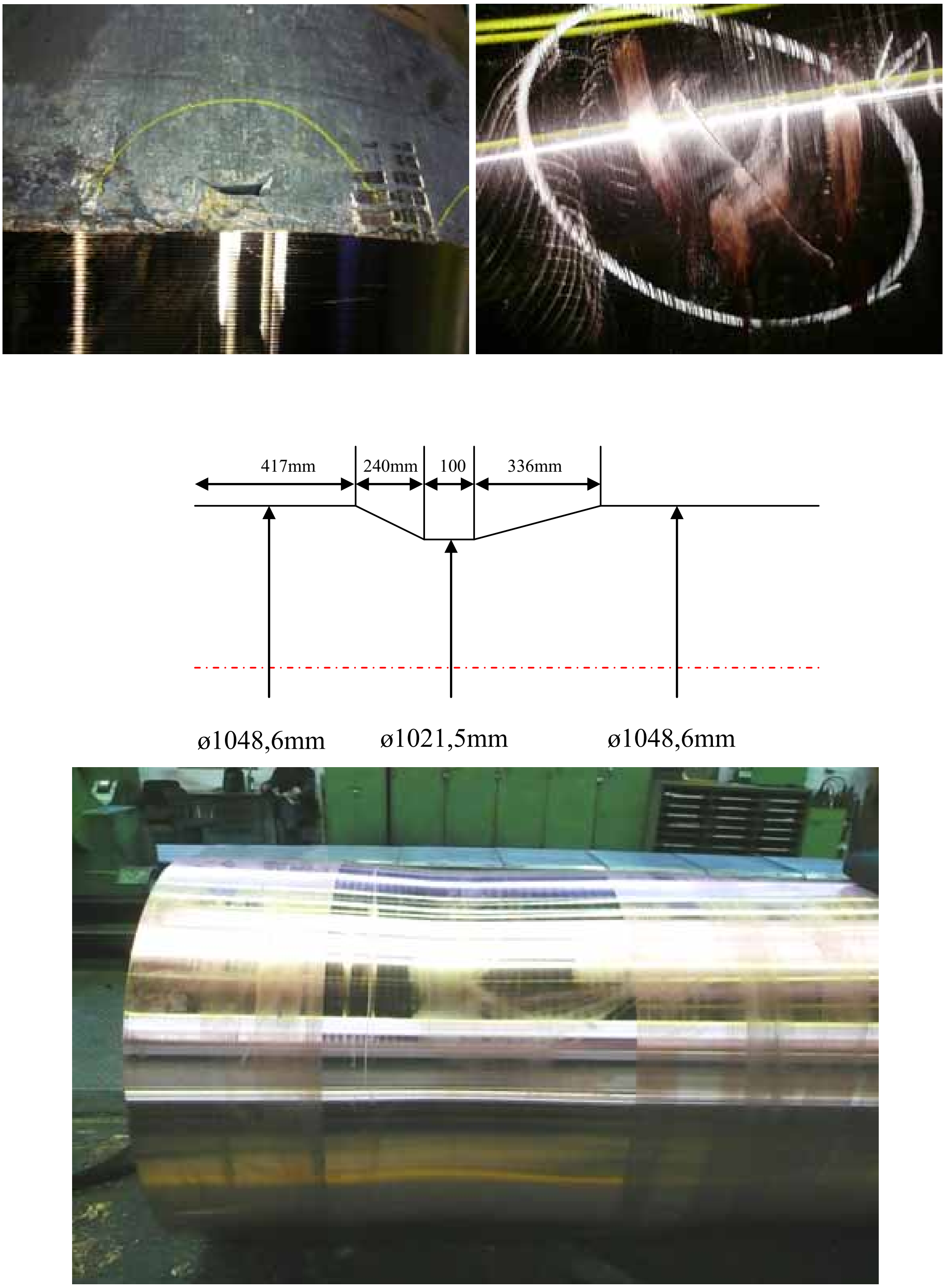
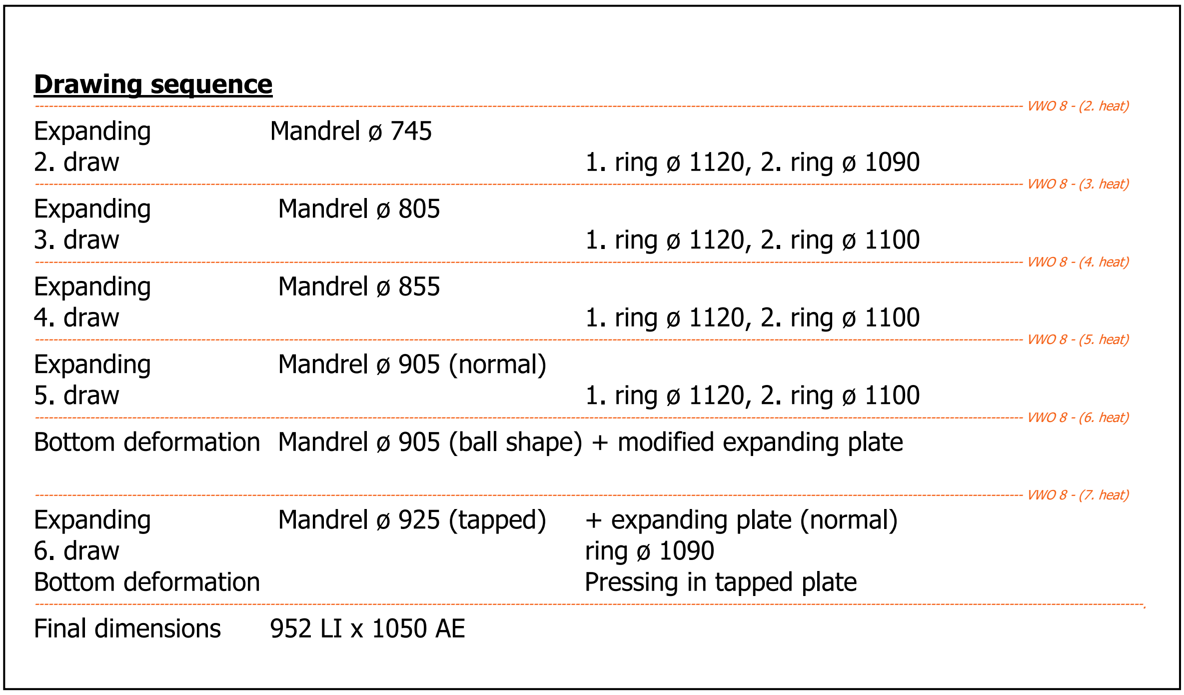


Figure 7. *The material faults in the top part of the T55, above. The machined cylinder after repair removal of the faults, below.*

3.3 Expanding and drawing steps

The deforming sequence after piercing was done for both T54 and T55 according to the sequence given in Figure 8. From the second draw to the fifth draw the outside of the cylinder was drawn through 2 rings, the first having a diameter of 1120 mm and the latter 1100 mm. After the fifth draw the bottom was deformed outwards with special mandrel, and after the sixth draw the bottom was reshaped with a specially formed blind ring back to planar form, see Figures 9 and 10.



Drawing sequence

			VWO 8 - (2. heat)
Expanding 2. draw	Mandrel ø 745	1. ring ø 1120, 2. ring ø 1090	
			VWO 8 - (3. heat)
Expanding 3. draw	Mandrel ø 805	1. ring ø 1120, 2. ring ø 1100	
			VWO 8 - (4. heat)
Expanding 4. draw	Mandrel ø 855	1. ring ø 1120, 2. ring ø 1100	
			VWO 8 - (5. heat)
Expanding 5. draw	Mandrel ø 905 (normal)	1. ring ø 1120, 2. ring ø 1100	
			VWO 8 - (6. heat)
Bottom deformation	Mandrel ø 905 (ball shape) + modified expanding plate		
			VWO 8 - (7. heat)
Expanding 6. draw Bottom deformation	Mandrel ø 925 (tapped)	+ expanding plate (normal) ring ø 1090 Pressing in tapped plate	
Final dimensions	952 LI x 1050 AE		

Figure 8. *Fabrication steps after piercing. Heating was made before each of the hot working steps but the first shown in the figure. The first step was made immediately after the upsetting and piercing having no intermediate heating. The last expanding was made with a modified tool, mandrel of ∅925 mm (Vallourec & Mannesmann Tubes).*

Figure 9. *The new deformation devise used for the bottom end deformation of the T55. (Vallourec & Mannesmann Tubes).*

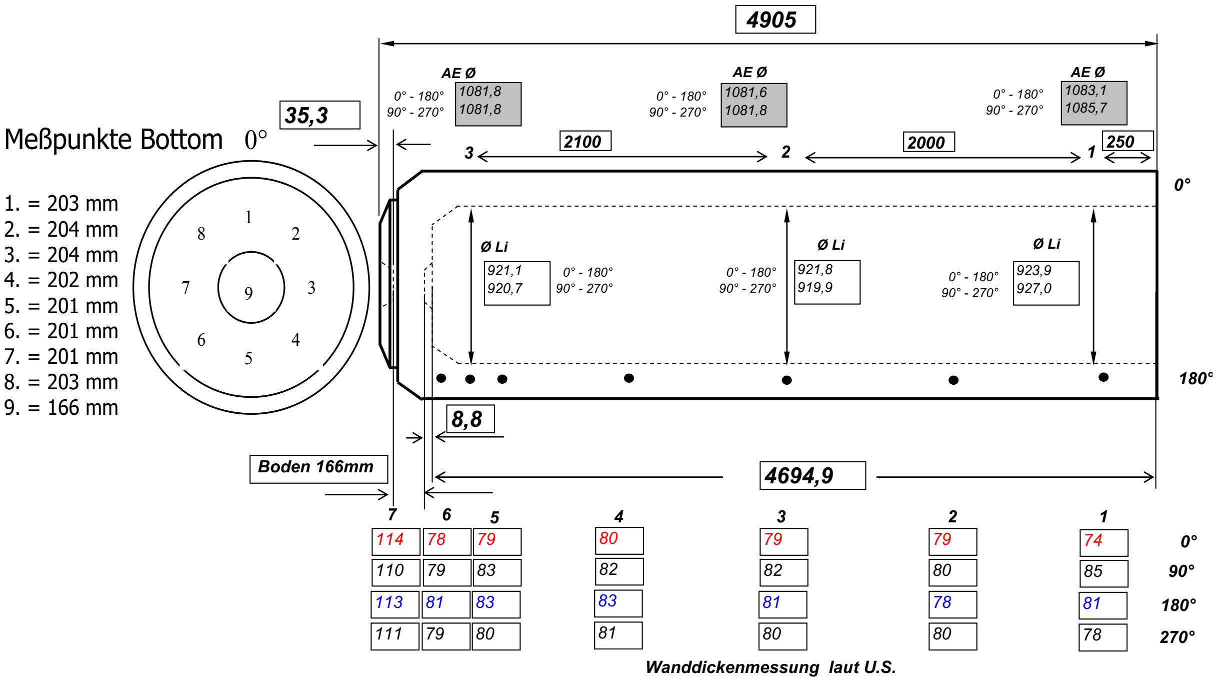
Figure 10. *Bottom was deformed after the fifth draw. On the left, T55 bottom after pressing with the spherical mandrel, and on the right, after deformation with the new device shown in Fig. 9. (Vallourec & Mannesmann Tubes).*

3.4 Special deformation of the integral bottom

Experience from earlier manufacturing tests has shown that the deformation rate was very often not enough in the centre of the bottom and in the corner area between bottom plate and the cylinder. The low deformation rate led to too coarse grain size in these locations.

Locally the deformation rate is increased with additional hot deformation steps with specially designed tools, half spherical mandrel and specially designed counterpart. And for the very central part of the bottom, an additional deformation device is used. The special deformation steps are shown in process description Figure 8 and the resulting shapes are shown in Figure 10.

After hot deformation process the T54 and T55 shells had dimensions according to Figures 11 and 12. The dimensions were acceptable for further manufacture.



	7	6	5	4	3	2	1
0°	114	78	79	80	79	79	74
90°	110	79	83	82	82	80	85
180°	113	81	83	83	81	78	81
270°	111	79	80	81	80	80	78

Figure 11. *The dimensions of T54 after hot deformation process.*

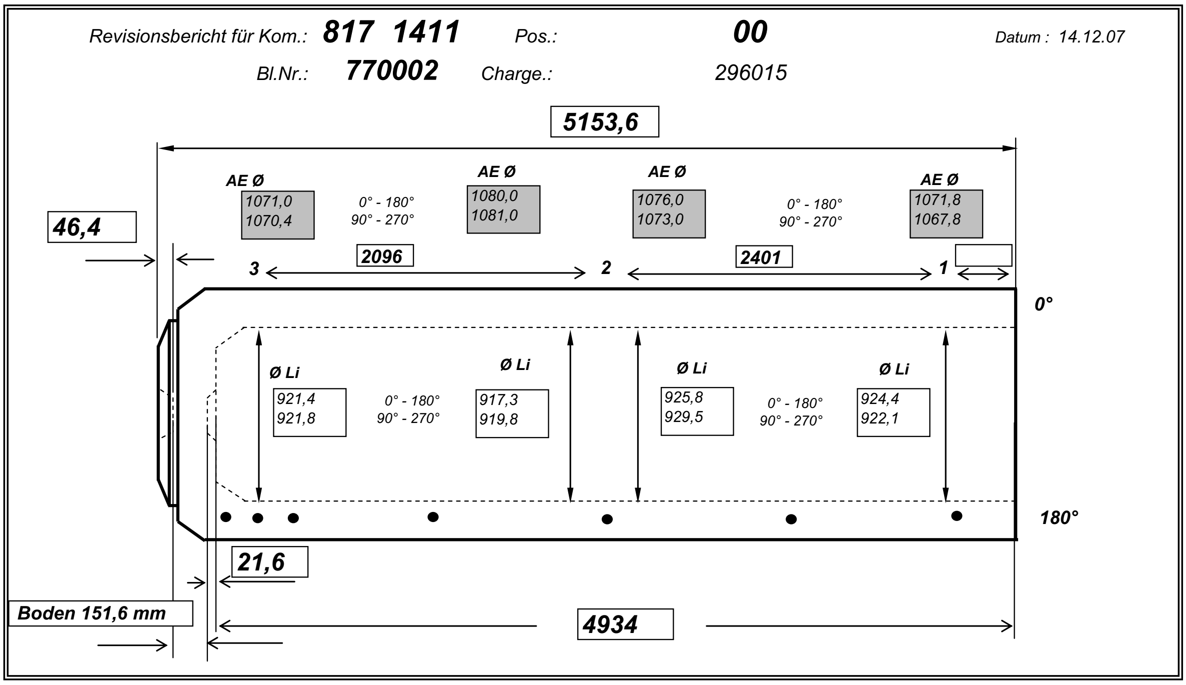


Figure 12. *The dimensions of T55 after hot deformation process.*

3.5 Process control

The process was controlled mainly by measuring the copper temperature before and after each deformation step. Between deformation steps the eccentricity of the cylinder was also controlled. Machining was needed to correct the eccentricity before continuing after the first pierce and draw step. The pyrometer measured surface temperatures before and after each deformation step for both of the ingots T54 and T55 are shown in Figure 13. The pressure in the press cylinders was also registered during various deformation steps. This gives information of the force and force marginal available during the hot deformation.

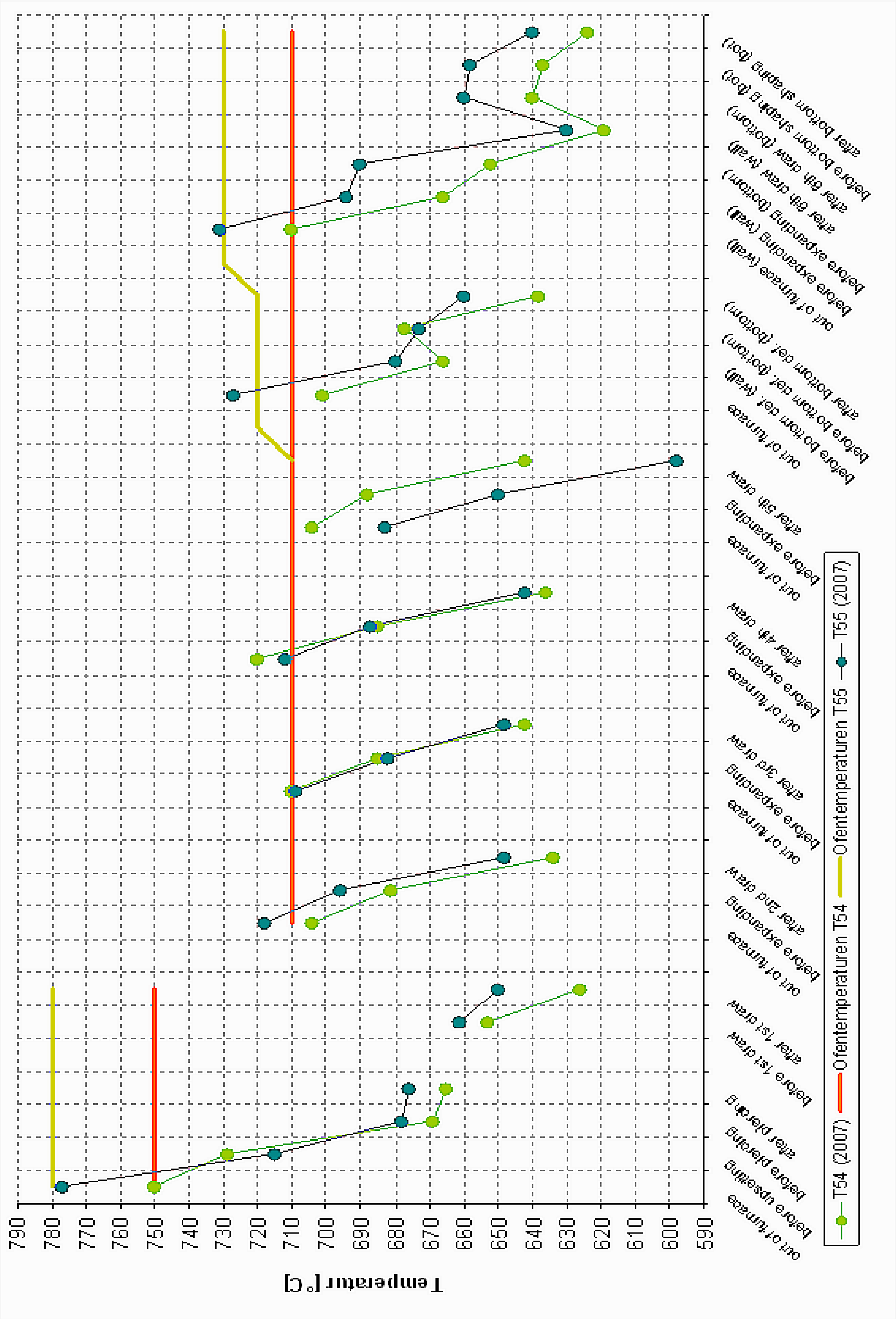


Figure 13. *The measured surface temperature before and after each hot working step. Light green colour corresponds to T54 and dark green to T55.*

4 MACHINING

The machining of the hot-formed tubes was made on Vallourec & Mannesmann Tubes mill in Reisholz. The shell was not machined as a whole. After hot deformation process, the bottom end, a ring from the middle of the cylinder and a ring from the top of the cylinder were cut off and transported to Luvata Pori Oy for destructive examination.

Then the two cylinders left from each of the shells T54 and T55 were machined for use as test material in the weld tests. Some of the copper shell parts and the bottom end plate of T55 are shown in Figure 14.

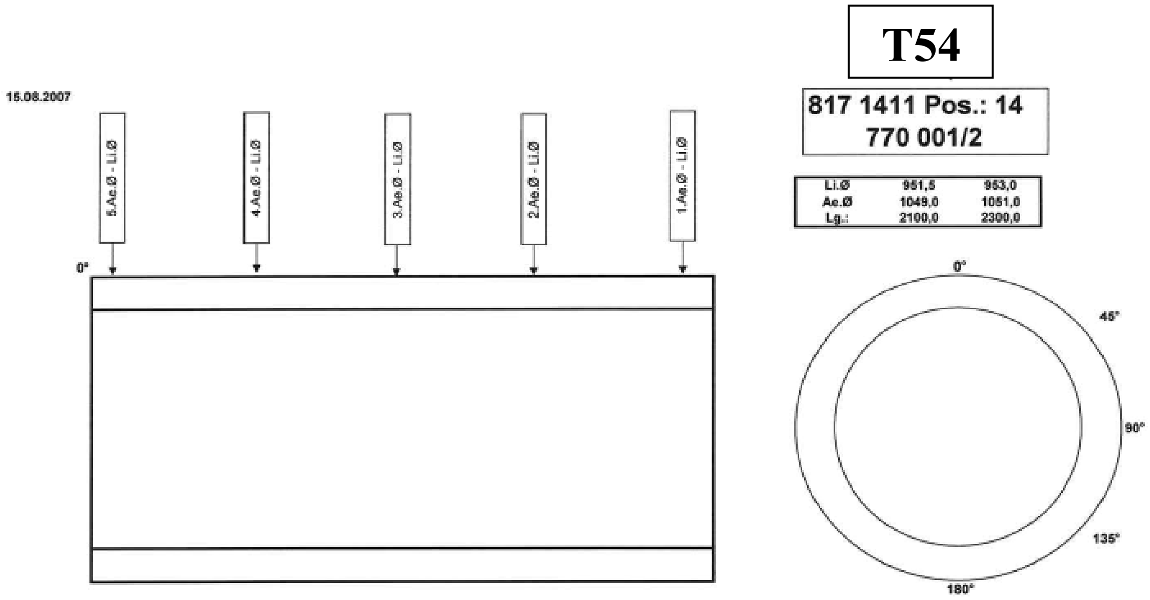
Figure 14. *The copper shell pieces from the T54 (upper) T55 (lower) after cutting the sample rings.*

5 CONTROLS

Vallourec & Mannesmann Tubes mill made the process control and the dimensional controls during and after the hot deformation process. After that the cylinders were cut into pieces and samples from bottom, middle and top of the cylinder were taken and they were transported to Luvata Pori Oy for further destructive inspections. The destructive control documents of Luvata Pori Oy are attached as Apps 6 and 7 at the end of this report.

5.1 Dimension control

The dimension control was made by controlling the main dimensions (length and diameter) of the copper shell from inside and from outside with 45 degrees interval. In addition, the cylinder wall thickness was controlled using ultrasonic device. The results are shown in Figures 15-17. The dimensional results fulfil the specified requirements.



Li.Ø	951,5	953,0
Ae.Ø	1049,0	1051,0
Lg.:	2100,0	2300,0

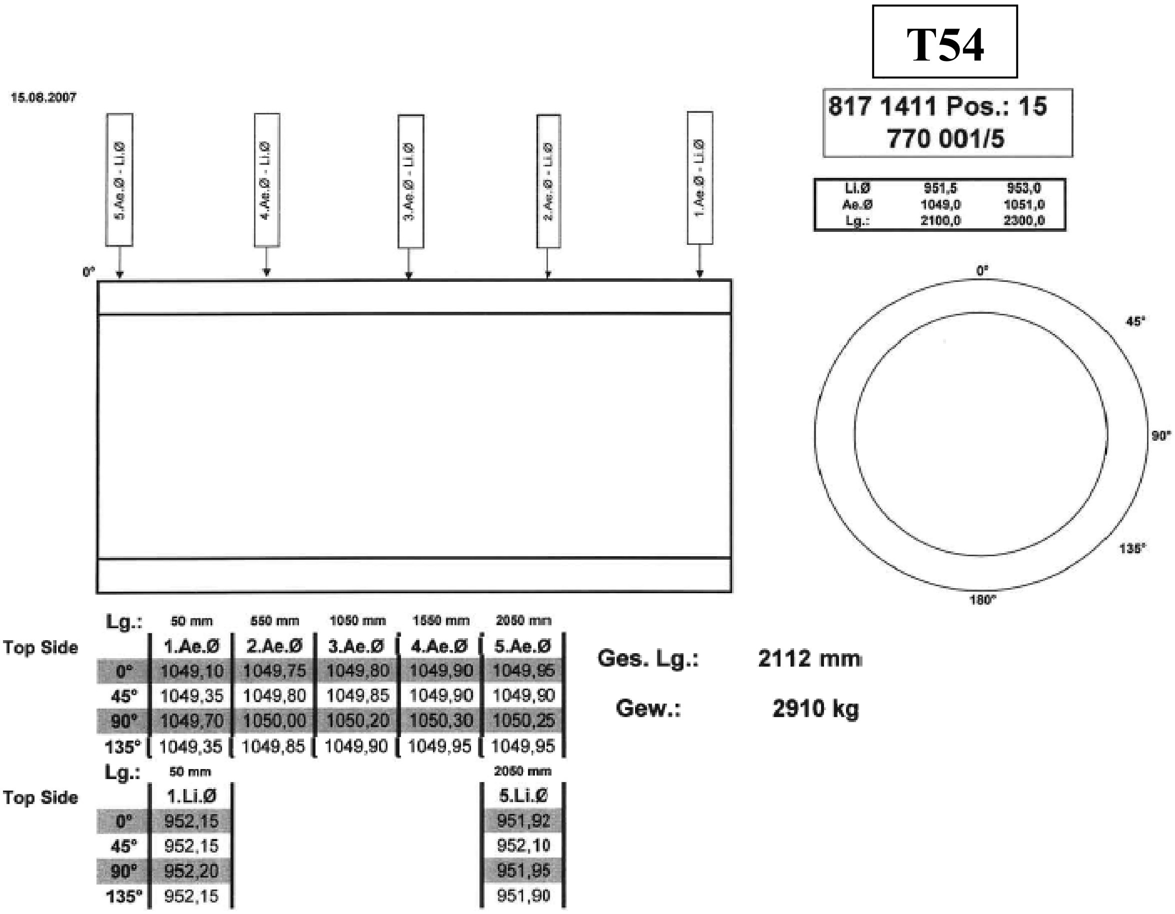
	Lg.:	50 mm	560 mm	1050 mm	1550 mm	2050 mm
Base Side		1.Ae.Ø	2.Ae.Ø	3.Ae.Ø	4.Ae.Ø	5.Ae.Ø
	0°	1049,85	1049,85	1049,90	1049,95	1050,00
	45°	1050,00	1049,95	1049,85	1049,75	1049,65
	90°	1049,90	1049,90	1049,95	1050,00	1050,00
	135°	1049,78	1049,83	1049,93	1050,05	1050,25

Ges. Lg.: **2119 mm**

Gew.: **2910 kg**

	Lg.:	50 mm	2050 mm
Base Side		1.Li.Ø	5.Li.Ø
	0°	951,80	952,20
	45°	952,20	952,05
	90°	952,00	952,05
	135°	951,70	952,15

***Figure 15**. The dimensional control results of the bottom part of the T54 cylinder after machining.*



Li.Ø	951,5	953,0
Ae.Ø	1049,0	1051,0
Lg.:	2100,0	2300,0

Lg.:	50 mm	550 mm	1050 mm	1550 mm	2050 mm
Top Side	1.Ae.Ø	2.Ae.Ø	3.Ae.Ø	4.Ae.Ø	5.Ae.Ø
0°	1049,10	1049,75	1049,80	1049,90	1049,95
45°	1049,35	1049,80	1049,85	1049,90	1049,90
90°	1049,70	1050,00	1050,20	1050,30	1050,25
135°	1049,35	1049,85	1049,90	1049,95	1049,95

Lg.:	50 mm	2050 mm
Top Side	1.Li.Ø	5.Li.Ø
0°	952,15	951,92
45°	952,15	952,10
90°	952,20	951,95
135°	952,15	951,90

Ges. Lg.: 2112 mm

Gew.: 2910 kg

***Figure 16**. The dimensional control results of the top part of the T54 cylinder after machining.*

5.2 Surface roughness control

Surface roughness control was not made in this phase, because the pipes were as raw material for EB-weld test.

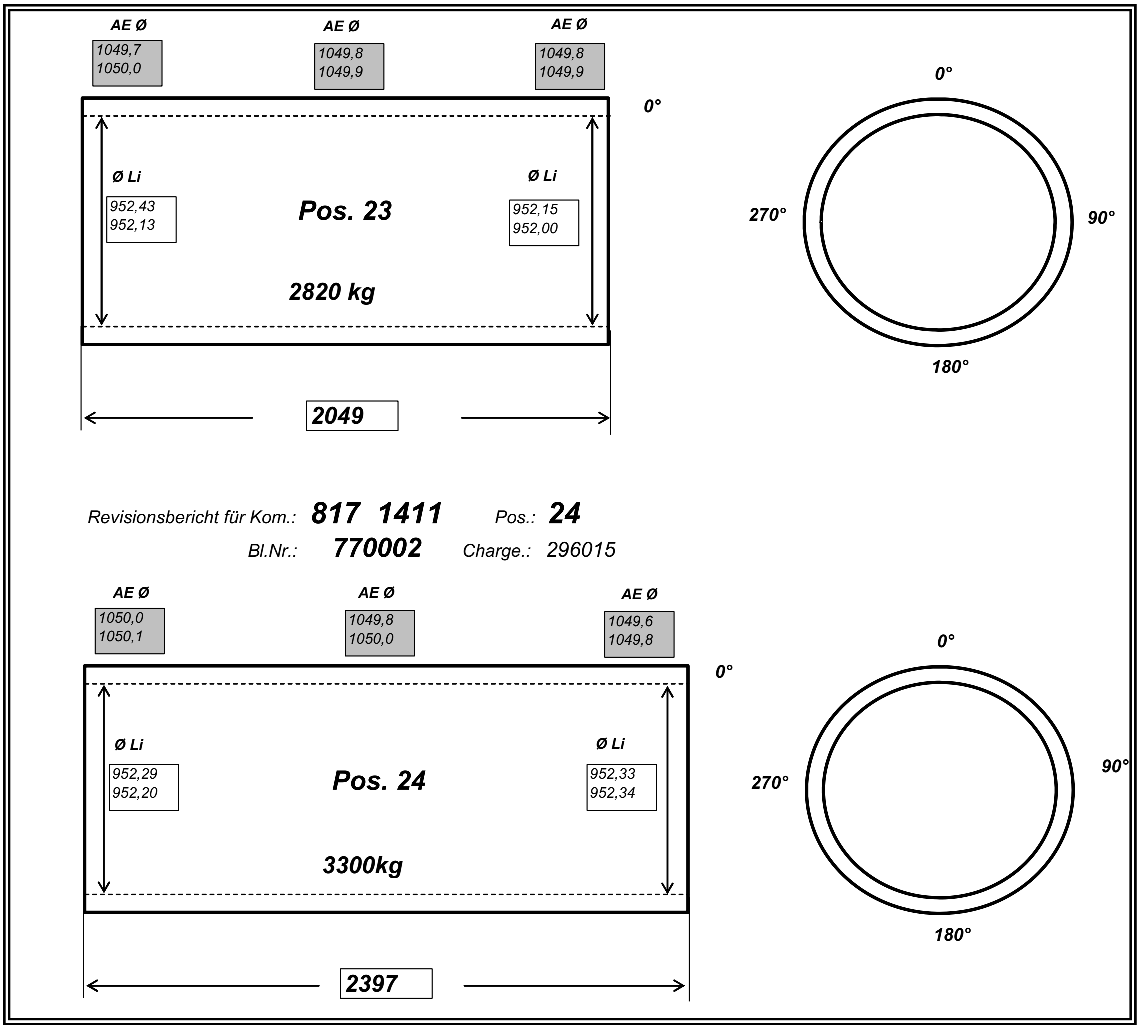


***Figure 17**. The dimensional control results of the parts of the T55 cylinder after machining.*

5.3 Ultrasonic control

Ultrasonic control was made for the machined pipes in SKB canister laboratory, Oskarshamn, Sweden, by SKB's and Posiva's NDT-examiners. The C-scan pictures of T54 and T55 cylinders ultrasonic examination are shown in Figures 18 and 19 for T54 and T55, respectively. The pictures give an indication of the material structure variations in various areas. C-scan presentations indicate damping variations of ultrasound in copper material that corresponds mainly to variance of grain size. However, the grain size variation is well within specification, which was also verified in the destructive tests, see chapter 5.4.2. No remarkable defects were detected in the pipe material. The amount of attenuation is depending on the frequency of the probe, which in these measurements was 5 MHz phased array probe. Higher damping area means normally area having larger grain size. In C-scan presentations the highest attenuation is areas where the colour is black or blue. Lowest attenuation is indicated with red colour. Vertical stripes in pictures originate from combining ultrasonic data files together but not having any indication of material changes caused by manufacturing.

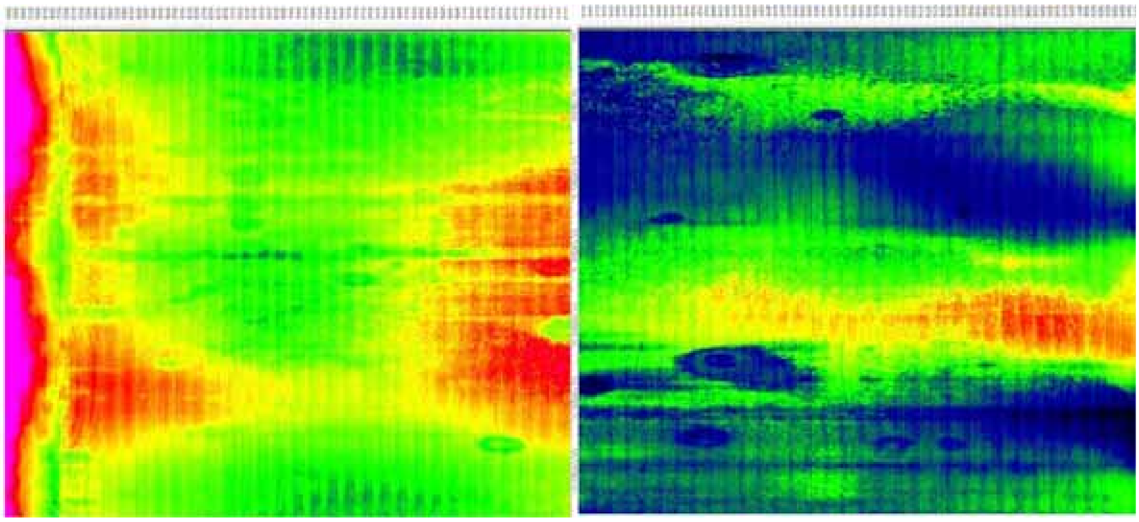


Figure 18. *C-scan presentations of ultrasonic testing of the cylinders of the T54 tube after machining.*

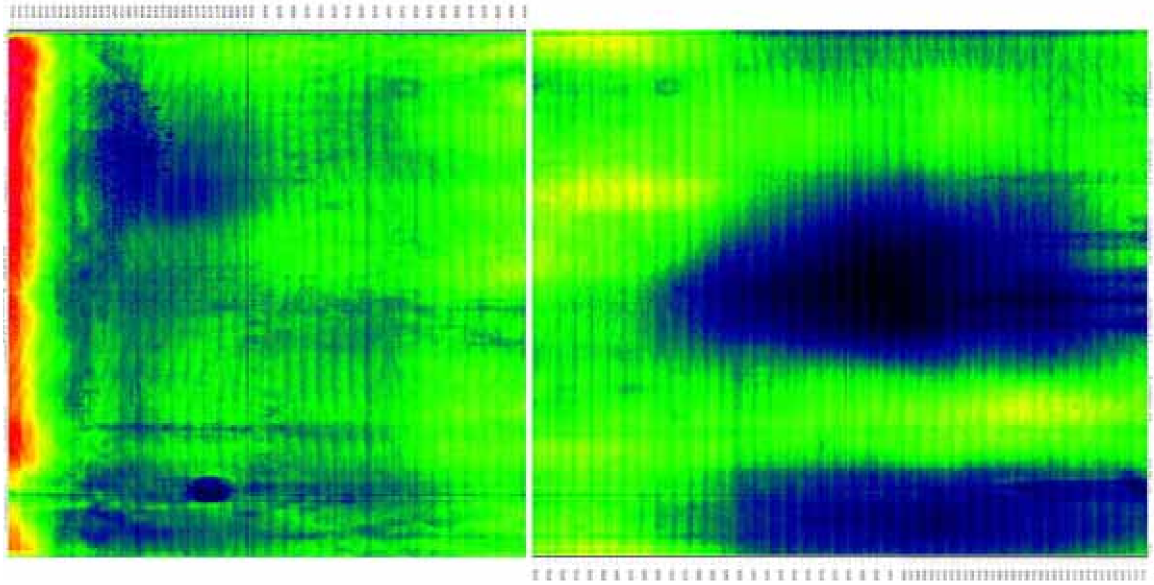


Figure 19. *C-scan presentations of ultrasonic testing of the cylinders of the T55 tube after machining.*

Colour code in Figures 18 and 19 is as follows: black or blue illustrates highest attenuation, red illustrates lowest attenuation.

5.4 Destructive controls

The two complete canister copper shells were cut into pieces as shown in Figure 1 of the Luvata reports (Apps 6 and 7) and these samples delivered to Luvata Pori Oy, for more precise examinations. The entire Luvata reports are attached as appendices to this report (Apps. 6 and 7).

5.4.1 Hardness testing

The hardness of the copper samples from T54 and T55 bottom plates and test rings was tested and reported in Luvata reports, Tables 1 and 2. The measured hardness values were between 36-40 HV and thus well below the acceptance limit of 65 HV.

5.4.2 Grain size testing

The grain size of the test rings and bottom plate of the T54 and T55 cylinders were investigated according to Luvata reports. The grain sizes are reported in Luvata report, Tables 1 and 2. The grain size was smaller than the specified maximum value 360 µm in all examined locations of the samples. Generally, the grain size was smallest at the centreline of the bottom, the greatest at corner area of the bottom end. The cylindrical part had grain size of 150 – 300 µm. In bottom end close to corner area there are some grains that are not completely re-crystallised.

5.4.3 Stress-strain testing

Tensile pieces for tensile testing performed according to EN 1563:1997 and tensile tests in accordance to EN 10002-1. The tensile test samples were taken from all the three test pieces in three orientations (circumferential positions of 0^o, 120^o and 240^o). The yield strength variation is 50-69 MPa for T54 and 44-47 MPa for T55. The tensile strength (206-210 MPa) and the ultimate elongation values (55-58 %) are very typical for soft copper. All elongation values fulfil with large margin the specified limit > 40 %. The results are presented in Luvata reports, Table 3, in Apps 6 and 7.

5.4.4 Chemical contents

Chemical composition was measured from two locations: top ring and base (0° -line) from both of the shells T54 and T55. Measured values fulfil all the specified requirements. The specified value of the micro-alloy phosphorus is 50 ± 20 ppm and the measured content varied only between 50-56 ppm. The measured contents of hydrogen, oxygen and sulphur were all remarkably lower than the maximum specified. All the control analysis results are presented in Table 6, in Apps 6 and 7.

5.4.5 Wall thickness measurement

The wall thickness of the shell T54 and T55 test rings was not applicable, because the samples were cut off before the cylinder was machined. The dimensional control results of the cylinders are presented in Figs. 15-17 in this report.

6 CONCLUSION

The results of the examinations showed that both of the test produced copper shells T54 and T55 fulfilled all the specified requirements as for soundness (integrity), mechanical properties, chemical composition, dimensions, hardness and grain size.

The canister tube T54 and the bottom was cut to pieces and investigated thoroughly at Luvata Pori Oy. There was significant improvement in grain size compared with the earlier results (T44 and T52). The grain sizes were clearly within the customer specification. However, some of the grains were not completely re-crystallised. These areas could be found on almost all of the investigated samples. The improvements made on site (Mannesmann, June 2007) to the extrusion process clearly improved the properties of the tube and the bottom.

Accordingly, the canister tube T55 and the bottom were cut to pieces and investigated thoroughly at Luvata Pori Oy. There was noticeable improvement in the grain structure of the tube wall compared with the results of the first manufacturing trial in this series (T54). The improvement was achieved by adjusting the process parameters at Mannesmann. The process adjustments were made to the piercing and final forging temperatures. The piercing temperature, however, was lower than anticipated due to the problems in lifting the blank.

The grain structure of CR-ring was almost optimal. The re-crystallisation degree was high and grain size variation was minimal. TR-ring had some larger grain regions. However, the specified limit for grain size was not exceeded. The grain size variation problem of the upper top part of the ring can be solved by cutting off more scrap from the top end.

The grain structure of bottom of the tube contained as-cast grain regions. The grain structure of the bottom of the tube is formed mainly during the piercing operation. Later operations have only minor effect on the structure of the bottom.

Vallourec & Mannesmann measurements of tubes T54 and T55 shows that there is difference in bottom thickness after piercing operation. The piercing depth of tube T54 was greater than of tube T55. For future experiments it is important to acknowledge the importance of adequate lubrication during the piercing operation and also the exact piercing depth, since it has major effect on the final grain structure of the bottom. One possible way to further deform the bottom of the tube is to pierce it further to make the bottom thinner. Thinner bottom can be deformed easier during the following processing steps as inner diameter of the tube is increased.

The latest improvements made on site (Mannesmann, December 2007) to the extrusion process of T55 improved particularly the properties of the cylindrical tube wall.

APPENDICES:

1. Quality plan for copper ingots, rev. 8 (2 pages)
2. Material certificate 07-74-2-1 (8 pages)
3. Material certificate 07-75-1-1 (8 pages)
4. Manufacturing and inspection plan, Vallourec & Mannesmann Tubes (2 pages)
5. The on-site supervision of T54 and T55 processing, Luvata Pori Oy (13 pages)
6. Testing of the base and the rings for the T54, 26.9.2007, Luvata Pori Oy (16 pages)
7. Testing of the base and the rings for the T55, 12.3.2008, Luvata Pori Oy (17 pages)

INSPECTION AND TEST ITEM	TEST METHOD AND DEVICE	SAMPLE SIZE/ FREQUENCE	SPECIFICATION OR STANDARD	CONTROL LIMIT ACCEPT. CRITERIA	WHO	RECORD	REMARKS
Acceptance of cathods	1 Chemical composition by spectrometer	Each cathode lot	ORC	Grade A+LME Reg.	ORC	Yes	
	2 Visual inspection	Each cathode lot	QCIM of Harjavalta Copper	Surface roughness	Harjavalta Copper / Foundry		
Acceptance of castings	1 Following of int. casting instructions and parameters				Foundry		
	2 Chemical composition by spectrom. and gas anal	Full anal. from top end.	SKB KTS001 Rev. 5	SKB order SKB KTS001 Rev. 5	Foundry	Yes	Hold point for release by MCP
	3 Visual surface inspection		QCIM of Foundry	Surface roughness and possible crack inspection	Foundry	Yes	Hold point for release by MCP
	4 Marking (identification)	Each ingot	Filling document: Final inspection, sampling/Ingot/SKB, rev 6	As per internal order between MCP and Foundry	Foundry	Yes	Written report
Final inspection and acceptance after rough machining	1 Visual and dimensional inspection		SKB order	SKB order	**MA**	Yes	Inspection report.
	2 Chemical composition by spectrom. and gas anal	Full anal. from both ends. Samples A, T and B each ingots.	SKB KTS001 Rev. 5 & Final inspection, sampling/Ingot/SKB, rev 6	SKB KTS001 Rev. 5	Met. lab./ ORC	Yes	EN 10 204 Mat. Certif.3.1.B
	3 Penetrant test for ends	Top end	EN 1371-1 (applied) EN 571-1	SP2-CP2 / LP 2c – AP 2c	**MA**	Yes	Inspection report.
		Bottom end	EN 571-1	Not yet specified	**MA**	Yes	(Both ends)
	4 Acceptance of 1-3		Filling document: Final inspection, sampling/Ingot/SKB, rev 6		**MA**		Hold point for release by SKB
Marking	Marking (identification)	Each ingot: top end (cast nro, SKB identify. nro.)	**SKB KT0705, Rev 5** Final inspection, sampling/Ingot/SKB, rev 6		**MA**	Yes	
Delivery permit	Written form SKB KTF07-7, rev 0	Each ingot	SKB KTF07-7, rev 0	Analysis, visual surface inspection, di-	**MA** / Customer	Yes	Hold point for release by

				mensional and penetrant test reports			SKB
Instruction for handling, packing and transportation	Must be followed during the whole manufacturing process	Each ingot	1.1.1.1 SKB KT0702, rev 5	Filling of Packing List: SKB Ingot Packing List, rev 0	**MA**	Yes	
Revision 8	**Issued by:** **Jouko Lammi**	**Date 24.05.2006**	Accepted by: **Pertti Mäkinen**	Date **24.05.2006**	Accepted by :		Date

Enclosure: **Document: FINAL INSPECTION, SAMPLING / INGOT / SKB Rev.7**
Document: SKB Ingot Packing List, rev 1

Quality Management system: **ISO 9001:2000, Det Norske Veritas Certificate No. 1757-2003-AQ-HEL-FINAS**

Luvata Pori Oy
Quality and Environment Services / HHA

EN 10 204 **INSPECTION**
24.04.2007 **CERTIFICATE 3.1**

Customer Posiva Oy	
Your order 9157-07	Our ref. LP 571640
Material Copper ingot diameter 850 mm SKB Spec. KTS001 REV.5	

Cast no.	B07-74-2-1, T07-74-2-1
Chemical composition	Appendix 1
Sampling picture and Dimensional control	Appendix 2
Visual and penetrant inspection	Appendix 3

LUVATA PORI Oy
These products have been produced under a certified quality system
ISO 9001:2000 / DNV Certificate No. 1757-2003-AQ-HEL-FINAS

We hereby certify, that the material described above complies with the order

Pauli Matt

Pauli Mattila
Author Inspector

APPENDIX I

ANALYSIS REPORT

CHEMICAL COMPOSITION

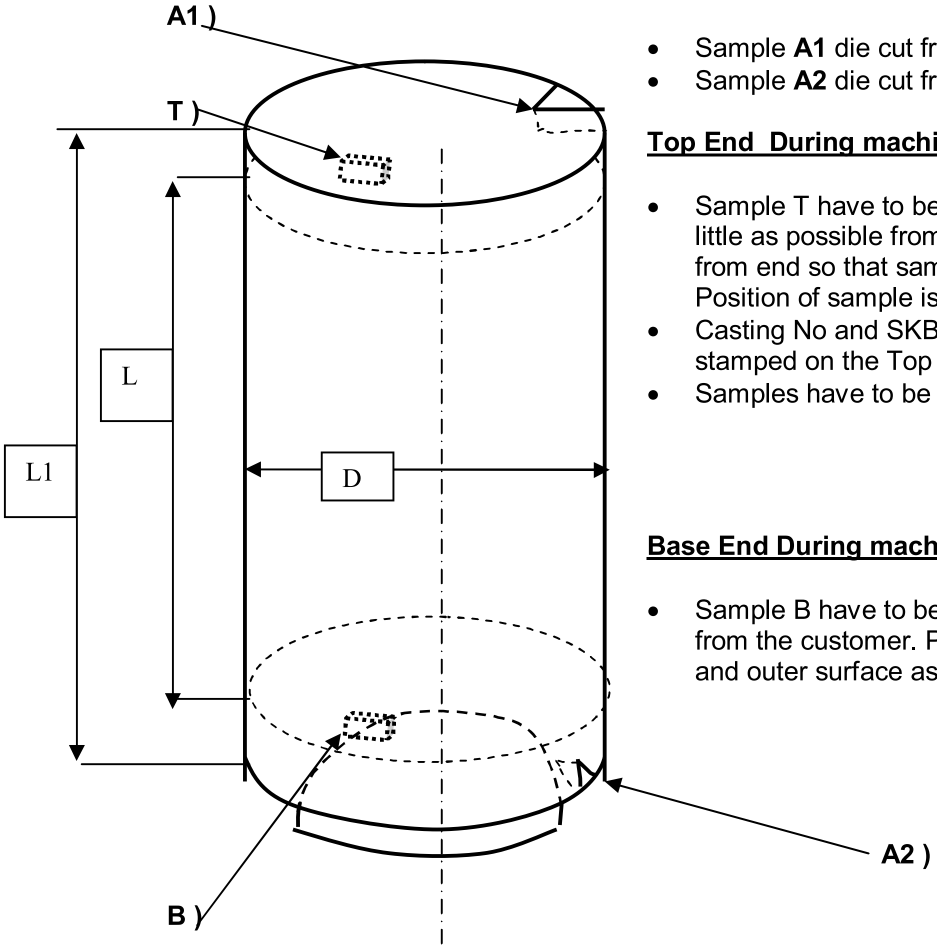
by spectrometer and gas analysator

		Spec.	Meas.	
Cast no			B07-74-2-1	T07-74-2-1
Cu	% min	99.99	99,992	99,992
Ag	Ppm max	25	13,1	13,1
As	“	5	0,83	0,82
Bi	“	1	0,13	0,13
Cd	“	1	<0,003	<0.003
Fe	“	10	0,5	0,3
H	“	<0.6	0,42	0,2
Hg	“	1	<0,5	<0.5
Mn	“	0.5	<0,1	<0.1
Ni	”	10	0,6	0,6
O	”	<5	1,9	1,0
P	“	30-70	53	53
Pb	“	5	0,32	0,32
S	“	<8	6,1	5,6
Sb	“	4	0,06	0,06
Se	”	3	0,1	0,2
Sn	”	2	0,06	0,07
Te	”	2	0,05	0,05
Zn	”	1	<0,1	<0.1

APPENDIX 2

FINAL INSPECTION, SAMPLING / INGOT/ SKB Rev. 7

Machining sample size ~10 x 40 x 40mm



Top End (Yp.) and Bottom End (Ap.) After casting

- Sample **A1** die cut from outer surface in foundry.
- Sample **A2** die cut from outer surface in foundry

Top End During machining

- Sample T have to be taken out by removing material as little as possible from end of ingots. 20-25 mm machining from end so that sample area is upraised from machining. Position of sample is between centerline and outer surface.
- Casting No and SKB identification number have to be stamped on the Top End
- Samples have to be stamped with the same numbers

Base End During machining

- Sample B have to be taken out according to the contract from the customer. Position of sample is between centerline and outer surface as close as possible from final length (L).

Penetration test and visual inspection have to be done for the surface before samples cut out by customer (MCP).

Posiva identification No:	Casting No:	Diameter Base End D_1	Diameter Top End D_2	Length of cast. L1	Weight of cast. kg	Length after machining L	Diameter after machining D	Weight after machining kg	SKB order requirement for weight, kg
T54	07-74-2-1	851	841	3070	15278	2828	827/830	13640	app. 13400

- **Sample A1 and A2 must be taken before delivery to machining shop. Dimensions and weight have to be filled in record (table above) and delivered this report to MCP.**
- **Sample T and B are final inspection samples. These samples shall be cut out from both ends of ingots and marked as T + casting nro: and B + casting nro: .**

24.05.2006/Jouko Lammi

23.04.2007 Jouko Lammi

VISUAL AND PENETRANT INSPECTION PHOTOS FOR THE ORDER
Posiva Oy No: 9157-07
CAST NO 07-74-2-1

Top of ingot after the 85mm end machining, Photo No 1141

Bottom of ingot after the 50 mm end machining, Photo No 1143

Bottom of ingot after the 100 mm end machining, Photo No 1144

Bottom of ingot after the 150 mm end machining, Photo No 1147

The ingot after the machining and grinding ready for delivery, Photo No 1719

Customer Posiva Oy	
Your order 9157-07	Our ref. LP 571640
Material Copper ingot diameter 850 mm SKB Spec. KTS001 REV.5	
Cast no.	B07-75-1-1, T07-75-1-1
Chemical composition	Appendix 1
Sampling picture and Dimensional control	Appendix 2
Visual and penetrant inspection	Appendix 3

LUVATA PORI Oy
These products have been produced under a certified quality system
ISO 9001:2000 / DNV Certificate No. 1757-2003-AQ-HEL-FINAS

We hereby certify, that the material described above complies with the order

Pauli Mattila
Author Inspector

APPENDIX I

ANALYSIS REPORT

CHEMICAL COMPOSITION

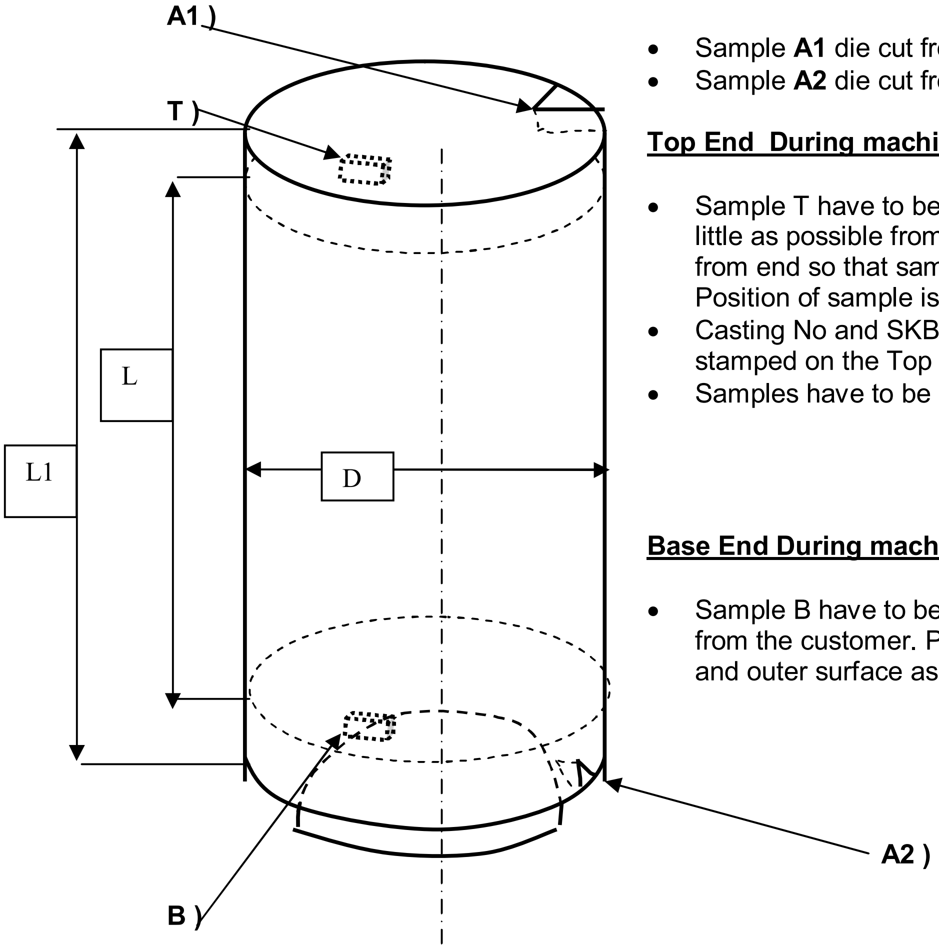
by spectrometer and gas analysator

		Spec.	Meas.	
Cast no			B07-75-1-1	T07-75-1-1
Cu	% min	99.99	99,992	99,993
Ag	Ppm max	25	12,7	12,9
As	“	5	0,84	0,85
Bi	“	1	0,14	0,14
Cd	“	1	<0,003	<0.003
Fe	“	10	0,3	0,3
H	“	<0.6	0,2	0,2
Hg	“	1	<0,5	<0.5
Mn	“	0.5	<0,1	<0.1
Ni	”	10	0,5	0,5
O	”	<5	2,0	3,3
P	“	30-70	54	51
Pb	“	5	0,36	0,35
S	“	<8	6,0	5,8
Sb	“	4	0,06	0,06
Se	”	3	0,2	0,2
Sn	”	2	0,07	0,07
Te	”	2	0,06	0,06
Zn	”	1	<0,1	<0.1

APPENDIX 2

FINAL INSPECTION, SAMPLING / INGOT/ SKB Rev. 7

Machining sample size ~10 x 40 x 40mm



Top End (Yp.) and Bottom End (Ap.) After casting

- Sample **A1** die cut from outer surface in foundry.
- Sample **A2** die cut from outer surface in foundry

Top End During machining

- Sample T have to be taken out by removing material as little as possible from end of ingots. 20-25 mm machining from end so that sample area is upraised from machining. Position of sample is between centerline and outer surface.
- Casting No and SKB identification number have to be stamped on the Top End
- Samples have to be stamped with the same numbers

Base End During machining

- Sample B have to be taken out according to the contract from the customer. Position of sample is between centerline and outer surface as close as possible from final length (L).

Penetration test and visual inspection have to be done for the surface before samples cut out by customer (MCP).

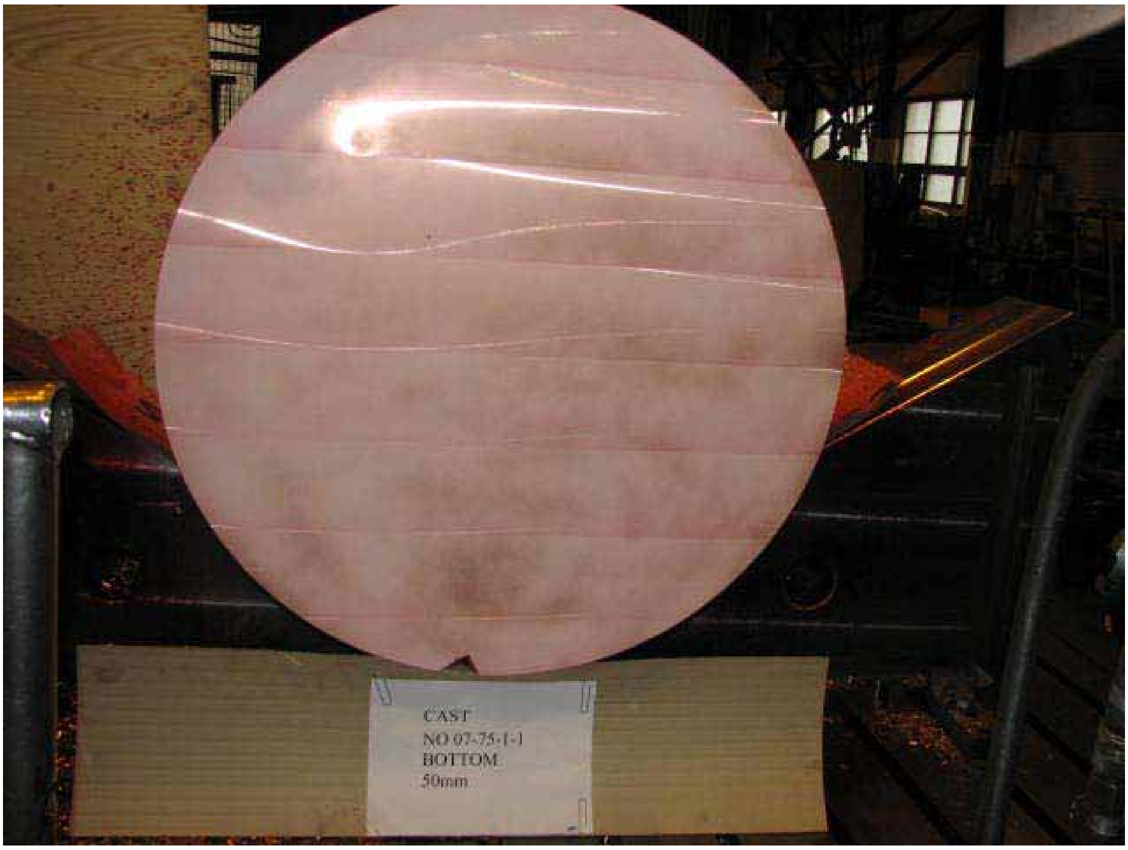
Posiva identification No:	Casting No:	Diameter Base End D_1	Diameter Top End D_2	Length of cast. L1	Weight of cast. kg	Length after machining L	Diameter after machining D	Weight after machining kg	SKB order requirement for weight, kg
T55	07-75-1-1	852	843	3110	15460	2792	830/834	13540	app. 13400

- **Sample A1 and A2 must be taken before delivery to machining shop. Dimensions and weight have to be filled in record (table above) and delivered this report to MCP.**
- **Sample T and B are final inspection samples. These samples shall be cut out from both ends of ingots and marked as T + casting nro: and B + casting nro: .**

24.05.2006/Jouko Lammi

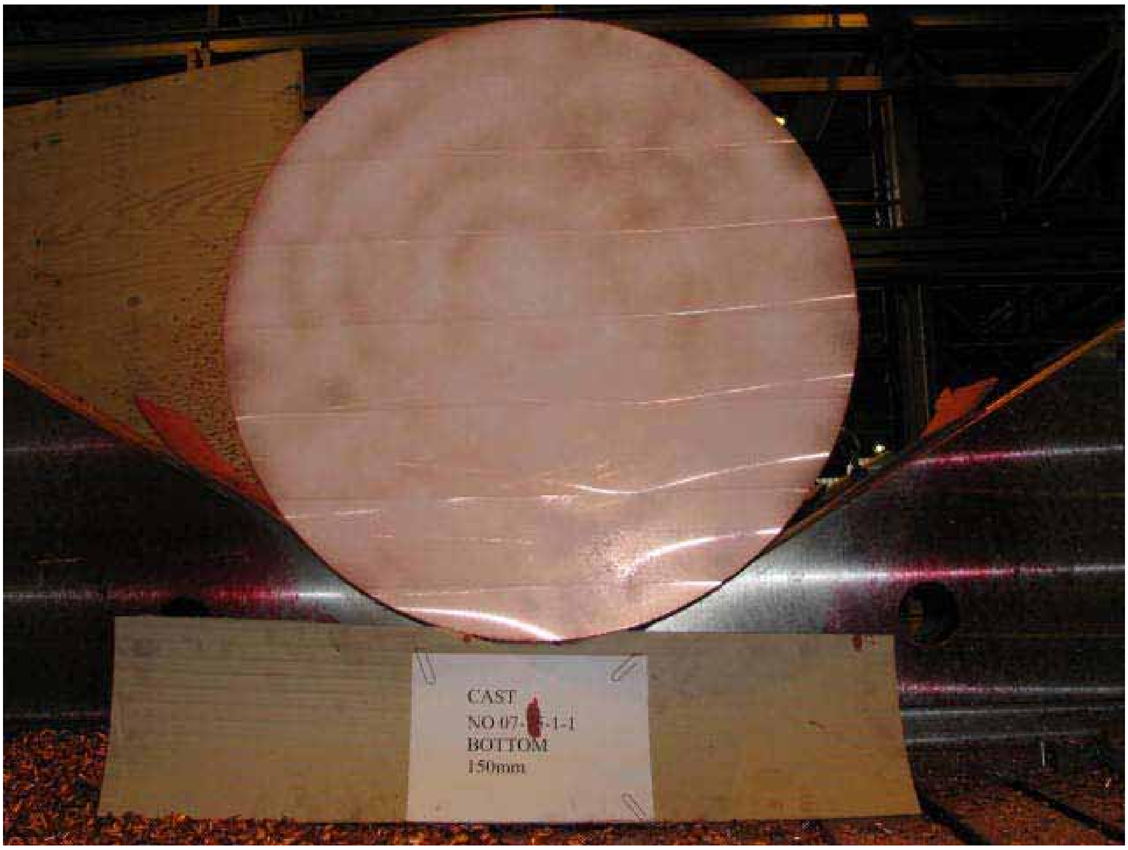
VISUAL AND PENETRANT INSPECTION PHOTOS FOR THE ORDER
Posiva Oy No: 9157-07
CAST NO 07-75-1-1

Top of ingot after the 85mm end machining, Photo No 1149

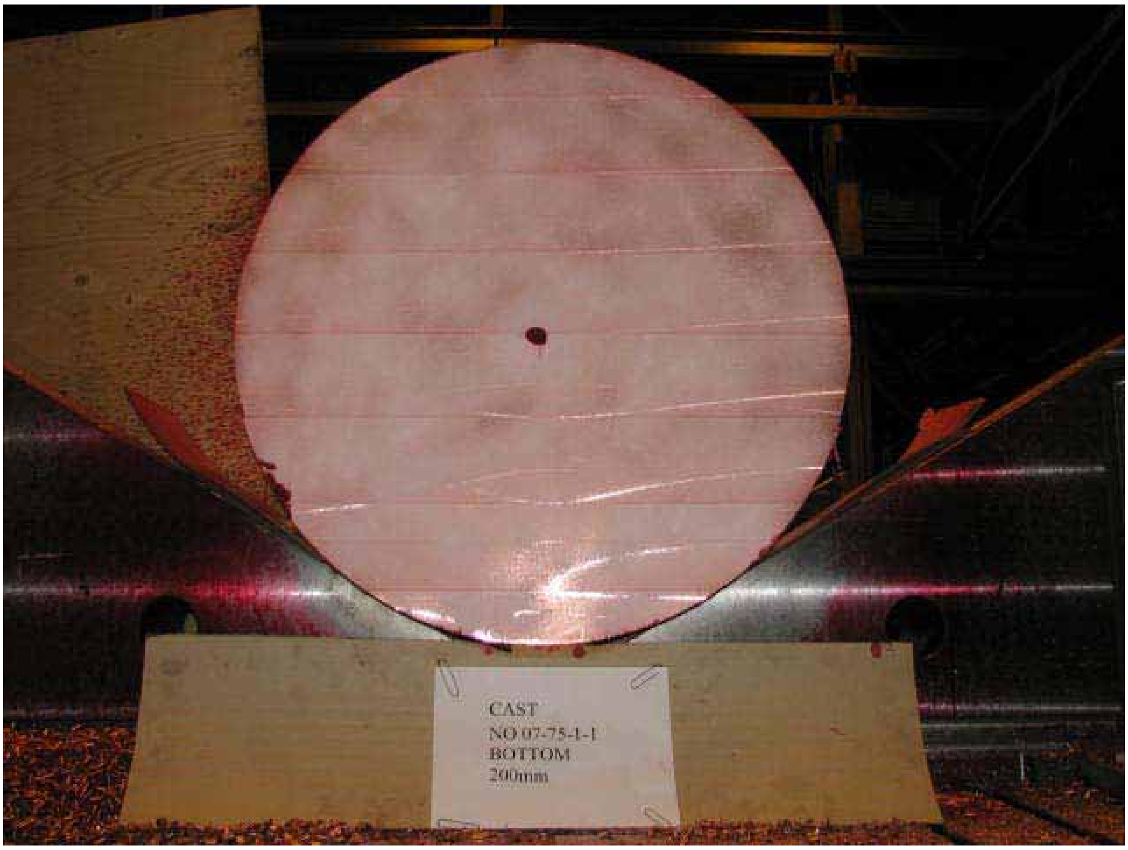


Bottom of ingot after the 50 mm end machining, Photo No 1151

Bottom of ingot after the 100 mm end machining, Photo No 1153



Bottom of ingot after the 150 mm end machining, Photo No 1155



Bottom of ingot after the 200 mm end machining, Photo No 1157

Bottom of ingot after the 230 mm end machining, Photo No 1158

The ingot after the machining and grinding ready for delivery, Photo No 1717

V&M VALLOUREC & MANNESMANN TUBES	**Plant** Reisholz	**Quality Plan No.** 1411

MANUFACTURING AND INSPECTION PLAN

Page 1 of 2

V&M Order No.	817/1347	**V&M Item No.**	11-15
Customer	Posiva Oy, Olkiluoto	**Cust. Item No.**	T 54 Pos. 11-15
		Cust. Order No.	9287-07
Purchaser		**Purch. Order No.**	
Specification	Specification KTS 002 Rev. 1, and Procedures KT 0701 Rev. 4, KT 0702 Rev. 5, KT 0703 Rev. 4, KT 0704 Rev. 5, KT 0705 Rev. 5, KT 0801 Rev. 3, KT 1102 Rev. 3, KT 1103 Rev. 3,	**Grade**	**Copper**

Revision **Date**	0 30.05.07				

Op. No.	Operation	Procedure Specification	TPI			Comments	Rev
1	Contract review	Customer order				V&M Sales department	
2	Billet inspection					Surface, analysis	
3	Prematerial inspection	Mill standard KT 0803, Rev. 1				Identification, quantity	
4	Production release						
5	Heating of ingots					Manufacturing sequence temperature (780°C), ≥ 8h lubrication with F 70 M-40 V	
6	Upsetting					One step 840 to 1135 mm	
7	Piercing					690 ID	
8	Drawing					With drawing lubrication F 26 A 1100 / 1080 x 680 *) No 6 to 8 without reheating	
9	Inspection of dimension and surface					including straightness, ovality, and eccentricity	
10	Turning					If necessary	
11	Heating up the hollow					Temperature > 50°C below No 5 Holding time 1-2 h, depending on the size	

V&M VALLOUREC & MANNESMANN TUBES	**Plant**	Reisholz	**Quality Plan No.**	1411
MANUFACTURING AND INSPECTION PLAN			**Page**	2 of 2

Revision	0						

Op. No.	**Operation**	**Procedure Specification**	**TPI**			**Comments**	**Rev**
12	Drawing steps 2-6 expanding ID and reducing OD to previous size with intermediate reheating and lubrication as no 8 1120 / 1090 x 745 *) 1120 / 1100 x 805 1120 / 1100 x 855 1120 / 1100 x 905 ball-shaping of the base 1120 / 1100 x 930 end-forming of the base					*) XXXX / YYYY x ZZZ means, that two rings are used in one drawing-pass	
13	Cutting to length acc. to sampling plan	Order no. 9655/05/TJAL 7.7.2005 Appendix 4					
14	Marking	KT 0705 Rev. 4					
15	Certificate						
16	Delivery Permit	KTF 07-7 Rev. 0					
17	Dispatch	Delivery Permit signed by Posiva KT 0702, Rev. 3 KT 0703, Rev. 3					

Abbreviations

H	**Hold Point**	Material/Operation cannot proceed without TPI presence. If TPI fails to attend, production can continue if confirmed with the customer.
W	**Witness Point**	TPI invited to witness a specific operation. If TPI fails to attend, production can continue.
M	**Monitoring Point**	TPI to review several operations on a sample basis. If TPI fails to appear, production can proceed.
R	**Review Point**	No invitation to TPI. Reports are available for verification.

LUVATA PORI OY

REPORT

Timo Välimäki / Research and development
Tuomas Renfors/ Metallurgical Applications

20.5.2008

Posiva

The on-site supervision of T54 and T55 processing

20.5.2008

Distribution:

POSIVA: Veli-Matti Ämmälä, Tiina Jalonen

LUVATA PORI: Jouko Lammi, Timo Välimäki

BACKROUND

Timo Välimäki from Luvata Pori participated in on-site supervision of tubes T54 and T55 processing in Vallourec & Mannesmann in Germany. The purpose of the on-site visit was to supervise and improve the manufacturing process at V&M. Present on site were also Jouko Lammi from Luvata, Veli-Matti Ämmälä and Tiina Jalonen (only T54) from Posiva and Magnus Johansson from SKB.

TUBE T54, PHASE 1

Hot forging process of tube T54

The following factors were closely supervised.

1. Temperature of the work piece before and after each process stage
2. Centricity of the tools
3. The centricity of the work piece in forging mould
4. Lubrication of tools
5. Forging speed
6. Forging force
7. The handling and transporting times of the work piece and duration of each process stage

Notes on swaging stage of Tube T54

Centralization of the swaging tools went generally ok. The tools were lubricated with water-soluble graphite paste. The bottom part of the tool was additionally lubricated with granular graphite-sulphur mixture (particle size 20-30 mm). The temperature of the work piece after removal from the heating furnace was 760ºC. The temperature followed the manufacturing plan and the temperature measurement was accurate.

The transportation of the work piece to the swaging tool went without problems. The work piece was centred to the mould by using separate tool (figure 1).

The swaging disc was placed on top of the work piece (billet). The centralization of the disc failed and was visually eccentric (over 8%, figure 2).

Figure 1. Centring tool for the work piece.

Figure 2. Eccentric mounting of the swaging disk.

The transportation of the swaging tool to the hydraulic press caused the billet to move inside the tool. The swaging temperature measurement went well. The pressure reading of the equipment during swaging operation was 280 bars. The operation took 15 seconds. The visual inspection of the swaging tool and work piece inside it was not possible after swaging.

The temperature measurement after swaging was unreliable.

Suggested improvements:

- Three point centring of the swaging disc
- Visual inspection of the tool and work piece after swaging should be arranged

Notes on piercing stage of Tube T54

The beginning of the piercing operation went reasonably well but as the operation proceeded, the piercing speed slowed down considerably. The pressure reading of the equipment during piercing operation was 290-300 bars (maximum value). The operation took 95 seconds.

Suggested improvements:

- Piercing temperature should be 20-30ºC higher to lower the equipment hydraulic pressure from maximum limit.
- The centricity of the work piece after piercing should be measured

Notes on first drawing stage of Tube T54

Temperature of the pierced work piece and munch was measured before drawing. Results are presented below.

Outside	670ºC
Inside	720ºC
Average	700ºC
Punch	500ºC

The punch was pushed inside the pierced work piece and the work piece was pushed through two drawing dies (figure 3). The operation took 20 seconds. Temperature was measured again 2 minutes after drawing. The results are presented below.

Bottom	600ºC
Center	660ºC
Inside	700ºC

Figure 3. The work piece after drawing (2 minutes after drawing).

CONCLUSIONS AND SUMMARY (T 54, 1.st PHASE)

Overall the processing route at Vallourec & Mannesmann is capable of producing tube that meets the requirements. The equipment function well and the hot forging speed is adequate to achieve desired structure. The temperature of the work piece during one process step changes no more than 40-50ºC.

The centricity of the work piece after swaging and piercing was not on the desired level. The filling of the mould after swaging is presented in figure 4 (taken from V&M report).

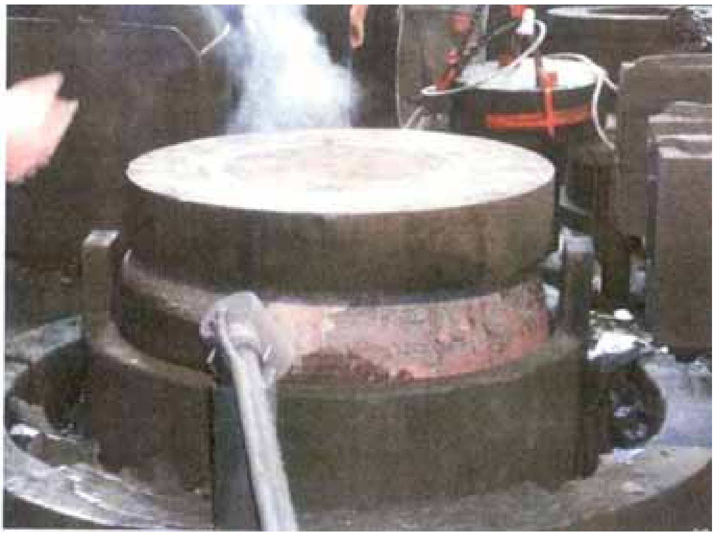


Figure 4. Uneven filling of the mould after swaging.

As figure 4 shows, the mould is not evenly filled after swaging. The left side of mould contains more material than right side. The piercing operation is very challenging to perform to billet so unevenly distributed inside the mould.

The piercing temperature of tube T54 was too low. The equipment was operating at its maximum force (pressure 300 bars). The work piece after piercing is presented in figure 5 (taken from V&M report.

Figure 5. The work piece after piercing.

As figure 5 shows, the pierced hole is not in the centre of the work piece. The wall thickness on the left side is significantly more than on the right side. The eccentricity traces back to the preceding stage, where the mould filling was uneven. As a conclusion, attention should be paid to the centring of the billet before swaging to enable even filling of the mould. This could be achieved for example by using three similar sized bars to centre the billet to the mould.

The punching-drawing is three-stage process step. Three separate hot deformations are applied to the material. The question is, whether the reduction on one step is high enough to recrystallize the whole structure.

TUBE T54, PHASE 2, (1.7.07- 3.7.07)

The wall thickness difference of the tube T54 (20 mm) after 1st phase had been machined to 5 mm. The second phase consisted of five separate increments of inner diameter of the tube with calibration of the outer diameter after each increment through two calibration dies (except the last one, which was pushed through one die). The diameters of the dies were the same during the first four id increment stages.

The second phase of the manufacturing process also contained separate stage for deforming the bottom of the tube with half round punch. The stopping block had also been shaped to deform the bottom.

Proceedings and measured results

During the second manufacturing phase special attention was paid to temperature measurement of the work piece, transfer times and forging equipment pressures. The measured results are presented in table 1 below.

1. Step				2. Step			3. Step		
	T	Time	Force	T	Time	Force	T	Time	Force
T_out of furnace	715ºC	(11.45)		725ºC	(14.05)		725ºC	(17.23)	
Push-draw	700ºC	5min	230 bar	690ºC	3min	285 bar	705ºC	4min	275 bar
T_end	670ºC	6min30s		675ºC	5min20s		695ºC	5min15s	
T_back in furnace	660ºC	14min		670ºC	11min30s		660ºC	10min	
4. Step				5. Step			6. Step		
	T	Time	Force	T	Time	Force	T	Time	Force
T_out of furnace	715ºC	(19.51)		710ºC	(21.23)		715ºC		
Push-draw	700ºC	3min30s	280 bar	690ºC	3min30s		705ºC		
T_end	685ºC	4min40s		670ºC	5min		695ºC		
T_back in furnace	645ºC	13min 20s		650ºC	10min30s		660ºC		

The process took total 11 hours to complete due to the long heating time of the work piece between each process stage (1h 30min). The forging press was also used for other purposes during the process.

Overall the push-draw steps went without problems and with adequate speed. The total cycle time of one step was 10-15 min. The hot deformation time of one step was 60-90 s. The temperatures were on the same level after removal from the heating furnace. The set furnace temperature value was 710ºC and the optical pyrometer readings were 0-15ºC above the set value. The most reliable location to measure the temperature was the tube bottom.

First bottom deformation (step 5)

The half round punch used for first deformation of the tube bottom is presented in figure 6.

Figure 6. The half-round punch.

The work piece after deformation is presented in figure 7.

Figure 7. The tube bottom after first deformation of the bottom.

As figure 7 shows, the lower end of the bottom has experienced some necking. The necking has occurred due to the mismatch of the shapes of the punch (half-round) and tube (flat).

Second bottom deformation (step 6)

The shape of the punch in second deformation of the tube bottom is presented in figure 8 and the shape of the end block is presented in figure 9.

Figure 8. The shape of the punch.

Figure 9. The shape of the end block.

The work piece after the second phase (after step 6) is presented in figure 10.

Figure 10. The work piece after step 6.

During the second deformation of the tube bottom (step 6) the half-round shape of the tube bottom was essentially flattened again.

The degree of deformation applied to the bottom of the tube is very limited. The most effective deformation is most probably applied to the bottom of the tube during the inner diameter increment steps (steps 1-4). As inner diameter of the tube increases, the bottom part is under tension, which deforms it.

TUBE T55, PHASE 1 (12.11.2007)

Some modifications were made to the manufacturing plan of tube T55 based on the results achieved with tube T54. The swaging temperature was increased from 760ºC to 790ºC. The granular graphite lubrication was left out and special attention was paid to centralization of the billet inside the swaging tool to improve the wall thickness uniformity after piercing.

The control measurements of the temperatures were conducted with optical pyrometer and results were documented by using the same method than with tube T54.

Notes on swaging stage of Tube T55

The transfer of the billet from the transportation cart and turning the billet to vertical position went without problems. The grip to the billet failed during the lifting to the swaging tool. The grip failed total three times and each time the billet was dropped to the ground from the crane.

The assumed benefit of the higher take-out temperature (790ºC) was lost during the lifting stage, since the lost of grip took extra time. In practice swaging was conducted in about the same temperature than tube T54.

The centralization tool had been modified after T54 to improve the centralization of the billet. The tool is presented in figure 11.

Figure 11. The centralization tool (tool id 855 mm, length of the straight part 400 mm).

The centralized billet inside the swaging tool is presented in figure 12.

Figure 12. The billet after centralizing.

The swaging disc was placed on top of the billet (figure 13).

Figure 13. Swaging disc on top of the billet.

The work piece after swaging is presented in figure 14 and after piercing in figure 15.

Figure 14. The work piece after swaging.

Figure 15. The work piece after piercing.

As figures 14 and 15 show, the work piece after swaging and piercing is lacking material on the left side. The centralization of the billet was not completely successful. The work piece after removing from the swaging tool is presented in figure 16.

Figure 16. The work piece after the phase 1.

CONCLUSIONS AND SUMMARY (T 55, 1.st PHASE)

Temperature after heating in furnace correlated well with the readings of optical pyrometer. The gripping problems in lifting the billet caused the temperature to sink below the anticipated level. The swaging temperature was at the same level than with tube T54. The anticipated improvements of the elevated temperature on deformation of the tube bottom and deformation speed were lost. The centricity of tube T55 after piercing was much better than of tube T54. The new centralizing tool was clear improvement. Overall the process parameters of phase 1 are comparable between T54 and T55.

TUBE T55, PHASE 2, (11.12.07- 12.12.07)

The second phase of tube T55 manufacturing turned out to be almost identical than of tube T54. The planned changes to the process parameters were mainly dropped due to the risk of necking of the tube. The only change in the manufacturing route was the temperature of two last push-draw steps. Temperature was raised 30ºC from 710ºC to 740ºC.

Proceedings and measured results

During the second manufacturing phase special attention was paid to temperature measurement of the work piece, transfer times and forging times. The measured results are presented in table 2 below.

1. Step			2. Step		3. Step	
	T	Time	T	Time	T	Time
T_out of furnace	755ºC	(12.04)	735ºC	(15.26)	725ºC	(18.26)
Push-draw	720ºC	3min11s	690ºC	3min40s	685ºC	3min
T_end	710ºC	1min39s	670ºC	1min	680ºC	1min20s
T_back in furnace	680ºC	12min10s	650ºC	11min	660ºC	14min
4. Step			5. Step		6. Step	
	T	Time	T	Time	T	Time
T_out of furnace	710ºC	(19.32)	745ºC	(9.43)	750ºC	(12.02)
Push-draw	680ºC	3min14s	700ºC	2min40s	715ºC	3min20s
T_end	655ºC	1min43s	700ºC	2min10s	710ºC	2min45s
T_back in furnace	580ºC	13min 15s	580ºC	6min30s	680ºC	2min

The first measured temperature with pyrometer (755ºC) differed significantly from the set furnace value (710ºC). The planned level of 710ºC was achieved before step 4. The conclusion of the temperature measurement was, that the difference in temperature reading is furnace related. Especially step 5 went much faster due to the higher hot deformation temperature. This elevated processing temperature should result in higher recrystallisation degree in bottom part of the tube.

Specific notes on the steps.

1.step) T_out 755ºC, significantly higher than with T54. There seemed to be lack of equipment force when the punch was pressed inside the tube. The punch was not lubricated. Temperature of the punch was 450ºC. V&M thought, that the work piece was too cold.

2.step) T_out 735ºC, the whole step went ok.

3.step) T_out 725ºC, the whole step went ok. The drawing die peeled material from the outer surface of the work piece.

4.step) T_out 710ºC, the whole step went ok. Temperature of the work piece decreased faster due to the increased surface area.

5.step) Equipment failure at 22.00. Temperature of the furnace was lowered to 400ºC. At 4.00 the temperature of the furnace was changed to 720ºC. The process step was restarted at 10.00 the following day. T_out 745ºC, the first bottom deformation to round shape, deformed better than T54 due to higher temperature.

6.step) T_out 750ºC, the bottom was straightened against end block. The diameter of the bottom increased during the operation. Push-draw through Ø1190 mm die was only barely successful because of increased temperature.

CONCLUSIONS AND SUMMARY (T 55, 2.nd PHASE)

Overall the 2nd processing phase of T55 went well even taking account the problems in controlling the work piece temperature. The furnace takeout temperature of the work piece had sinking trend during the first four steps. The planned temperature increment during the last two steps was successful. This change had favourable effect on the recrystallisation of the tube wall and deformation speed of the bottom.

The extra heating time after equipment failure (step 5) should not affect the grain structure of the work piece. Temperature should be much higher (about 900ºC) for secondary grain growth to occur.

During tube wall deformation steps some material was peeled off from the outer surface of the work piece. The removed material got stuck to the open (upper) end of the tube. Removal of the excess material was hard and time consuming. Even after the removal some excess material remained on the open end. There is a risk, that this extra material will be transferred inside the tube during push-draw operation. The material will become embedded to the internal wall of the tube and will cause defects. The sharp edge in the drawing edge may cause the material peel-off.

The key to control the process better is to verify the temperatures after the heating furnace. This can be achieved for example by installing four thermocouples directly to the work piece surface.

The handling and lifting of the work piece should also be improved.

LUVATA PORI OY
Timo Välimäki / Research and development
Tuomas Renfors/ Metallurgical Applications

REPORT

26.9.2007

Posiva

Testing of the base and the ring for the T54

26.9.2007

Distribution:

POSIVA: Veli-Matti Ämmälä, Tiina Jalonen

LUVATA PORI: Jouko Lammi, Timo Välimäki

INTRODUCTION

Nuclear canister tube T54 and tube bottom was received for structural inspection. The purpose was to determine grain sizes and grain size distributions of both bottom and tube wall. Mechanical properties and chemical compositions were also determined. Results from these studies are presented in this report.

Sample preparation

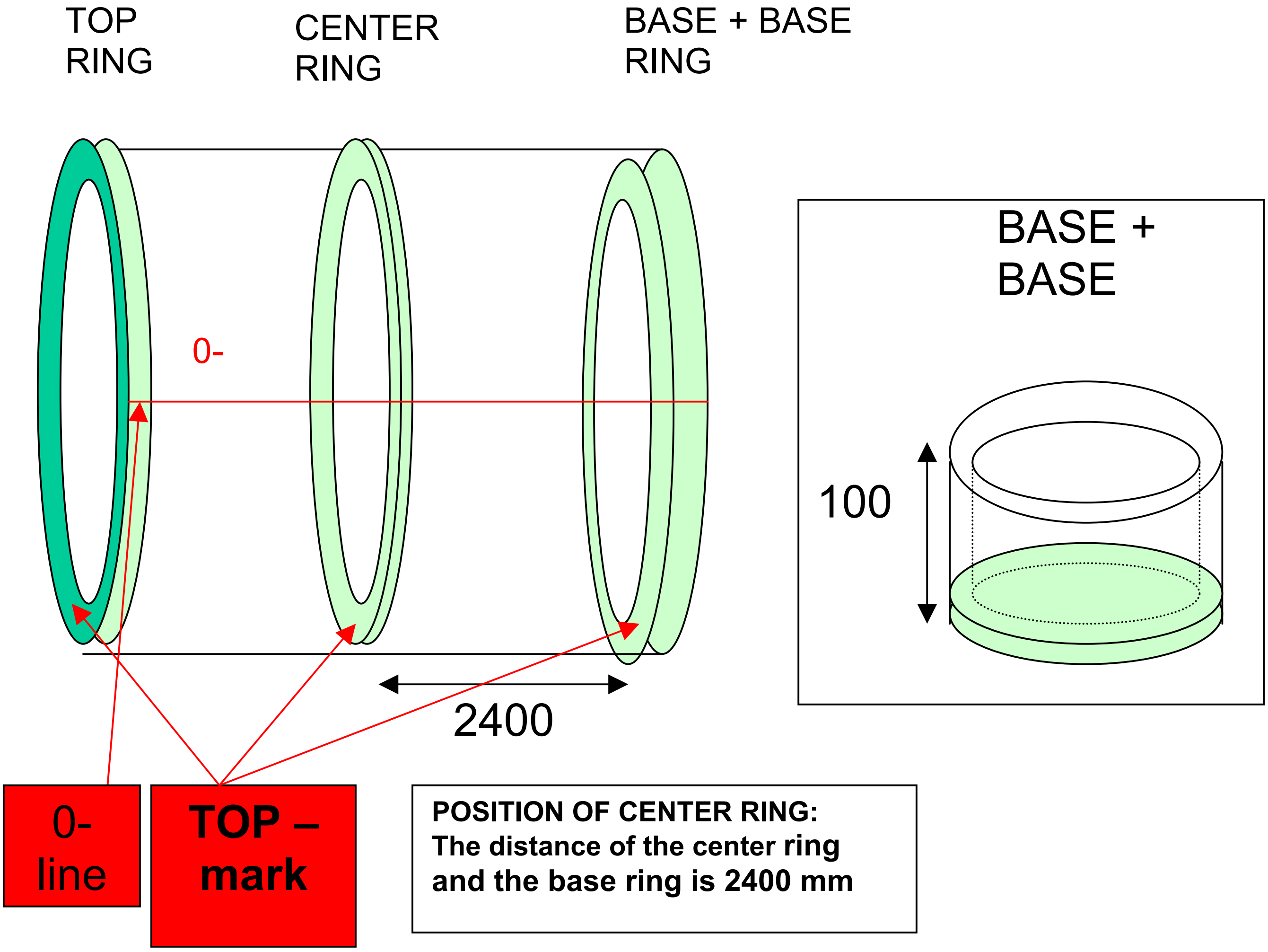
First the base was cut in half in two steps along the main diagonal (clockwise from 90° to 270°). Then the 25 mm slice was cut out from the base as we can see in Figure 1. After that, ¼ -slice (thickness 25 mm) was cut out from the base (clockwise from 180° to center) for more precise examinations.

From the top ring, centre ring and the base ring 250 mm length sectors were cut out around the spots 0°, 120° and 240° (clockwise). Afterwards these sections and the base slices were examined in Luvata Pori according to separate instructions (in finnish).

T54 SAMPLING – Marks (on red)

TOP AND CENTER RING:
The width of the ring
(along the tube axis)
is 50 mm

BASE AND BASE RING:
The base will be cut off at
(50 mm base thickness + 50 mm
ring) = 100 mm
from the outer surface of the
base



POSITION OF CENTER RING:
The distance of the center ring
and the base ring is 2400 mm

Figure 1. Sample locations

INSPECTION OF THE TUBE BOTTOMS

T54 tube bottom

An etched macro slice from T54 base is shown in Figure 2. As figure 2 shows, the base is not machined to final dimensions. The outer diameter of the base is ~1085 mm, inner diameter ~925 mm and the thickness of the base is 172-182 mm. Schematic picture in Fig 3 shows coarse grain areas in the bottom. Grain structures are shown in figures 4 to 8. Coarse grained areas are visible in the base corners (Figs 2 and 6) and both sides of the centreline (Figs 5 and 7), while the grains at the centreline are rather small (Fig 6).

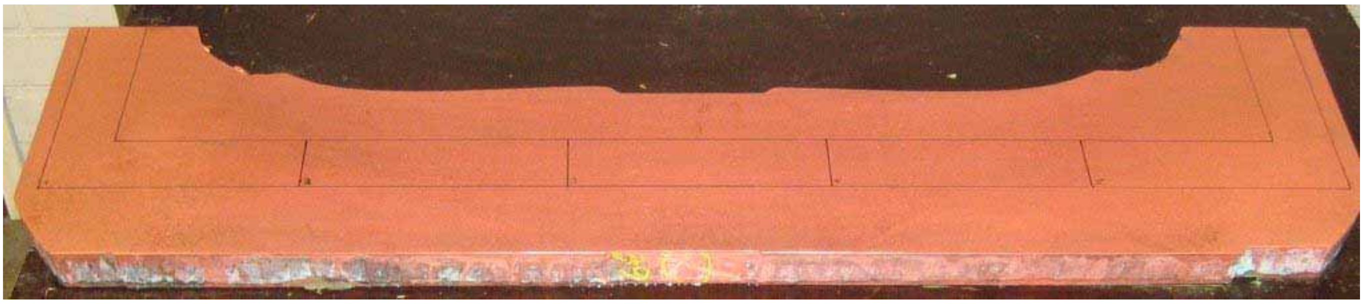


Figure 2 An etched base slice from T54 with sections drawn.

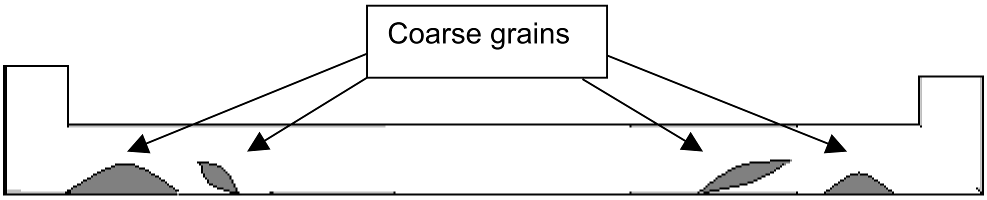


Figure 3. Coarse grain areas highlighted with grey.

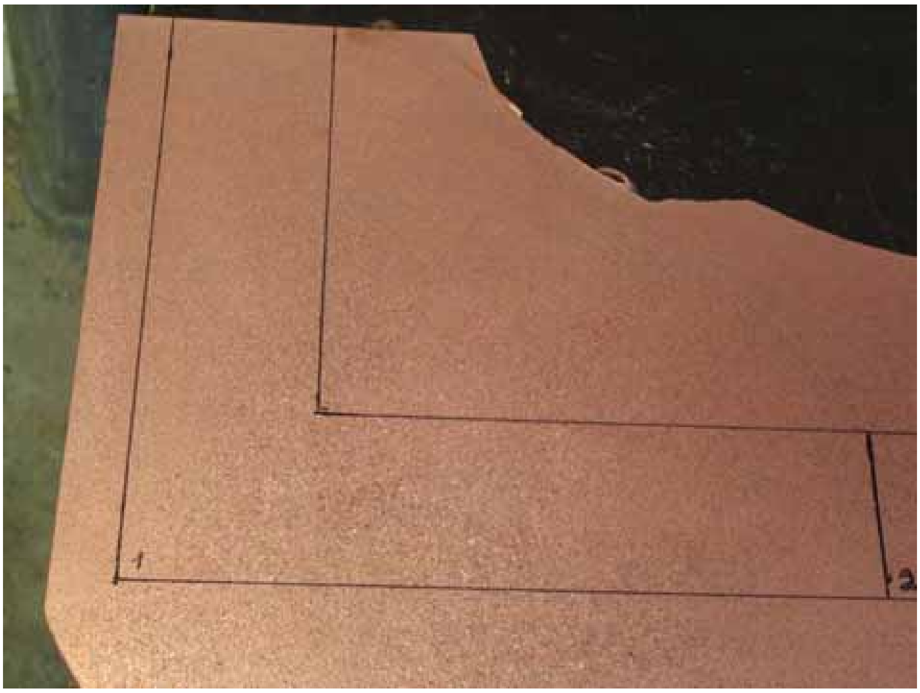


Figure 4. Corner area from the left hand side

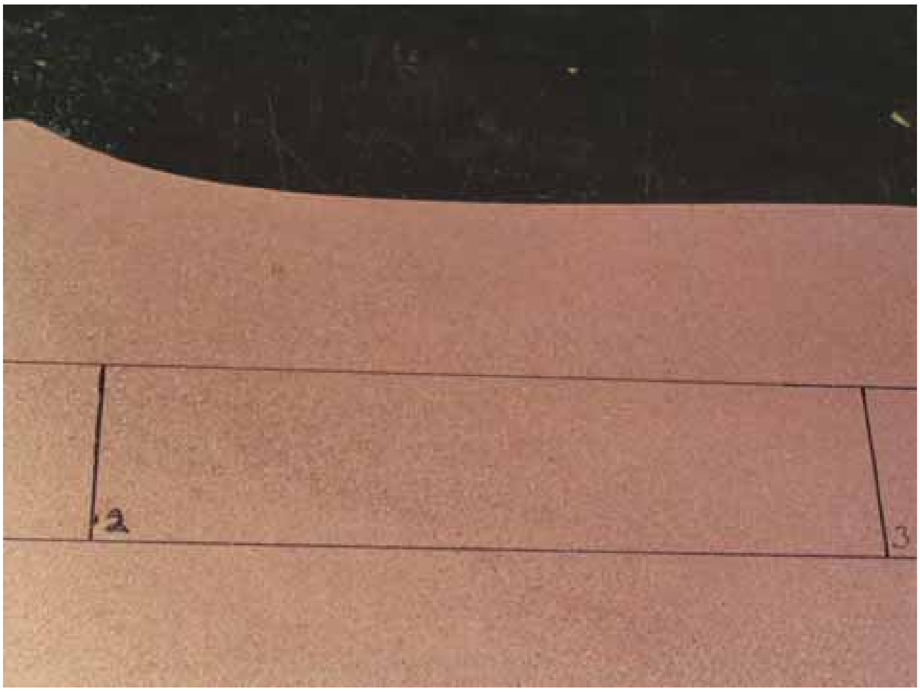


Figure 5. Microstructure from the area right next to left corner.

Figure 6. **Center of the tube bottom.**

Figure 7 Microstructure from the area right next to right corner.

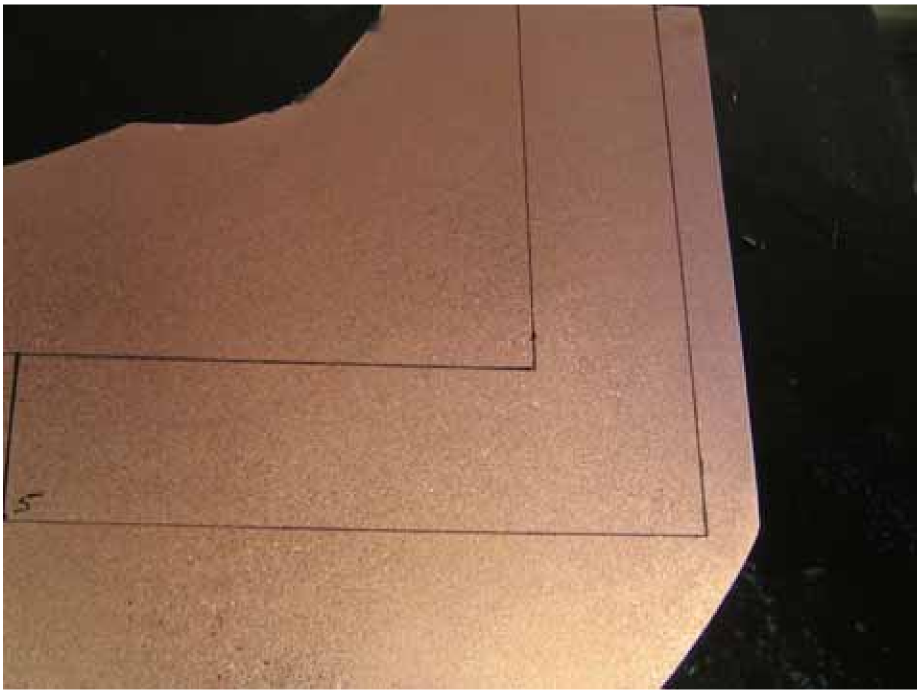


Figure 8. **Corner of the tube bottom on the right hand side.**

Hardness and grain size:

The ¼ -slice (thickness 25 mm) was cut transverse to the base slice. The ¼ -slice was cut into pieces as shown in Figure 9. Hardness and grain size examinations were made to both top and bottom side of the slide. Precise examination points are 140 (A), 210 (B), 290 (C), 370 (D) and 450 (E) mm from the outer diameter a.k.a in every 80 mm.

The grain size variation is 0,150-0,270 mm and results from the top and the bottom of the sample are close together. The grain size in the corner area is 0,220-0,270 mm. Grainsize becomes smaller towards the centreline (0,170-0,200 mm) and at the centreline the grainsize is at its smallest, 0,150 mm.

The hardness variation is 37-40 HV and essentially the same in bottom and top. Hardness and grainsize results are shown in Table 1 and Figures 10 and 11. All samples are well below the specified hardness limit (<65 HV) and grain size limit (<0,360 mm).

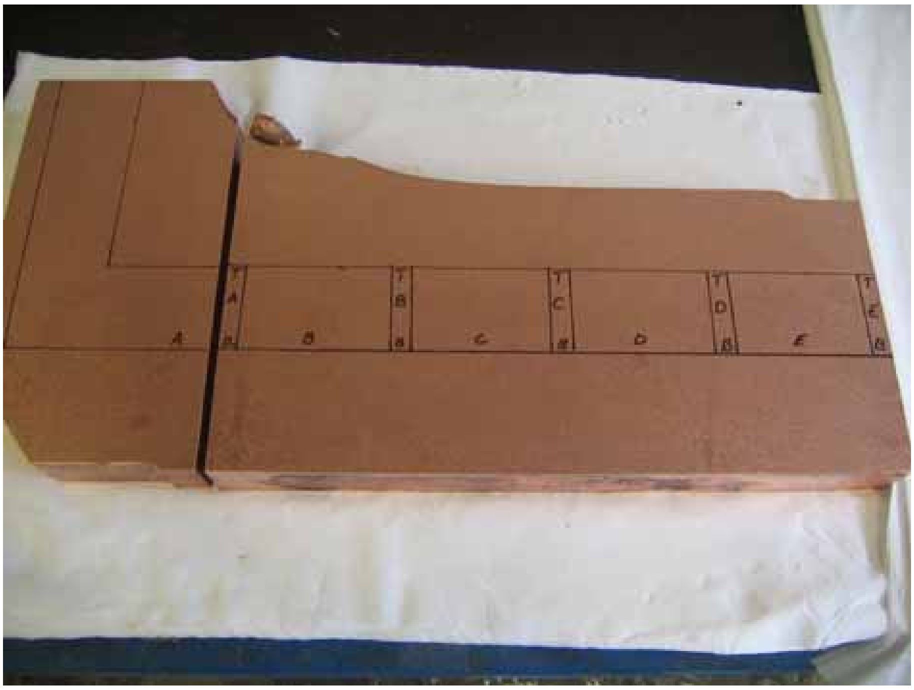


Figure 9 Sample locations for hardness and grain size measurements

Table 1. The hardness and grain sizes of T54 canister bottom along the inside radius every 80 mm towards to centre.

	Hardness [HV5]		Grain size [mm]	
	TOP	**BOTTOM**	**TOP**	**BOTTOM**
A	39	40	0,220	0,270
B	38	38	0,170	0,200
C	37	37	0,200	0,170
D	37	38	0,200	0,200
E	37	37	0,150	0,150

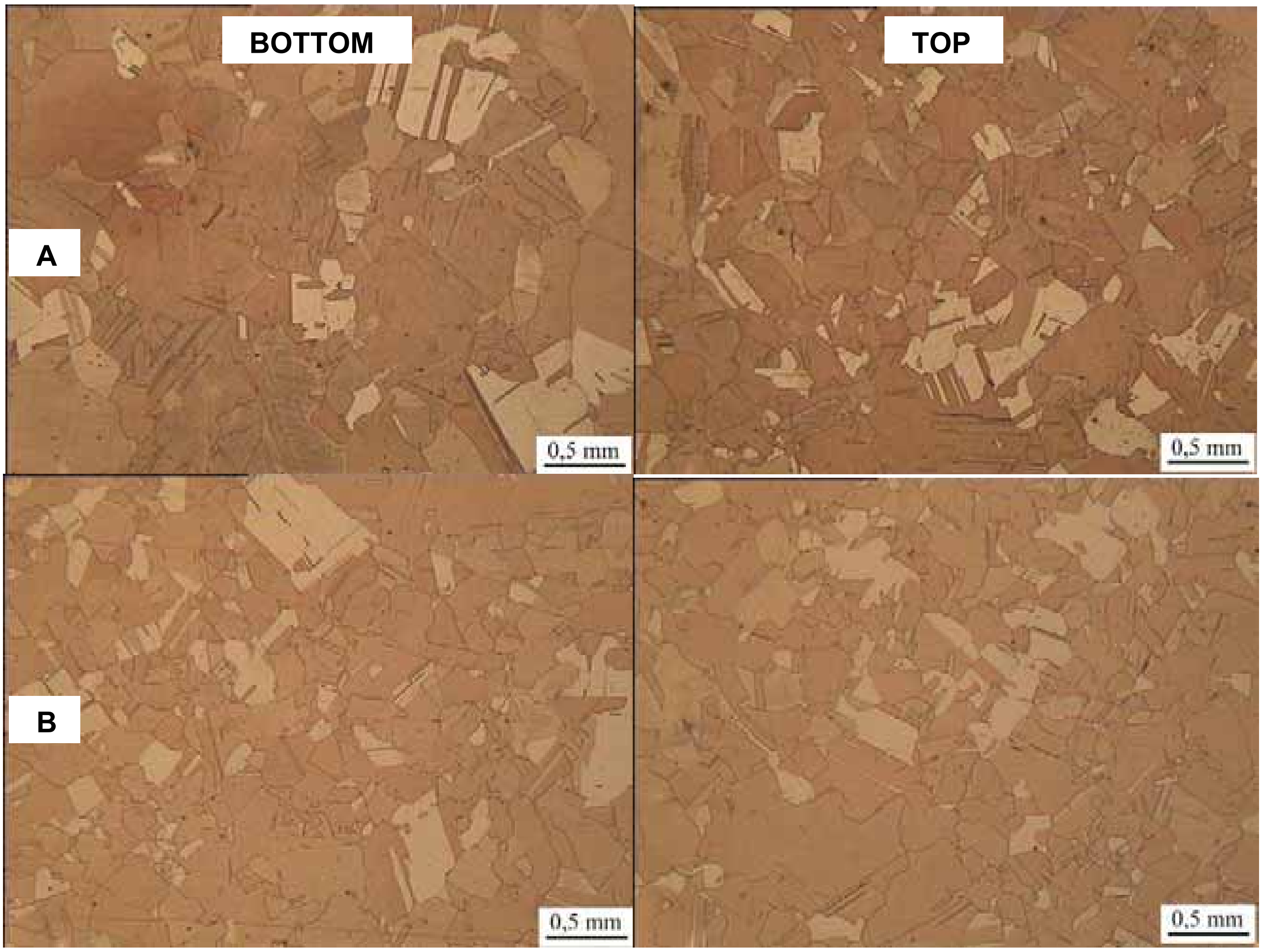


Figure 10 Microstructure from T54 sample A (upper - bottom/top) and B. 32x

Figure 11. Microstructure from sample C (upper - bottom/top), D and E. 32x

Some of the grains are not completely recrystallised. These areas can be found on almost all of the investigated samples (figures 10 and 11).

INSPECTION OF THE RINGS

The sectors for the examination were cut out from the rings at three circumferential locations 0°, 120° and 240° (Figure 12). The length of the sectors is about 250 mm and the rings are specified as Top Ring (TR), Center Ring (CR) and Base Ring (BR). Samples for tensile tests were taken from the inner circle of the ring. Samples for hardness and grain size measurements were taken from the outer circle of the ring. The locations of the samples are shown in Figure 13.

Figure 12. The sectors from the rings were cut out as above.

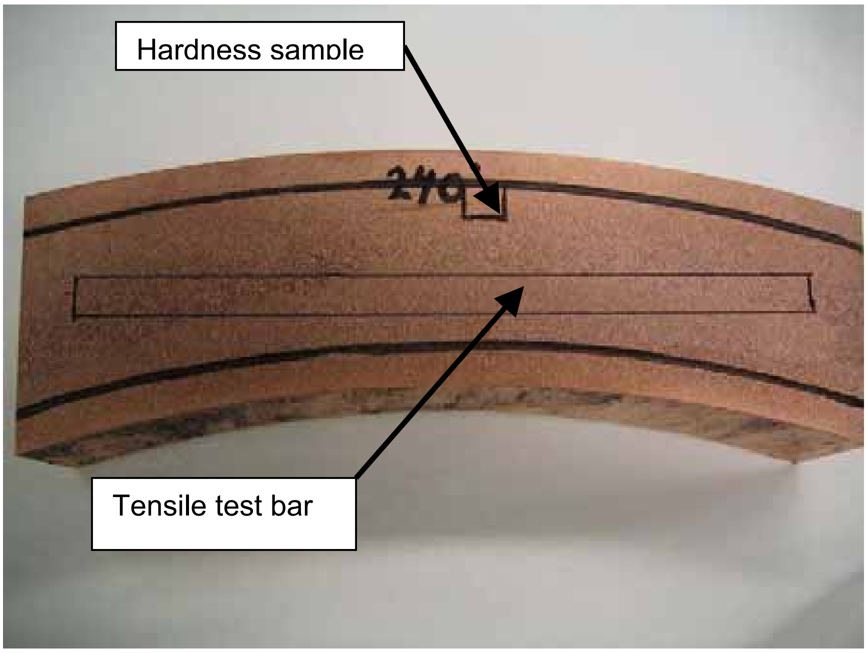


Figure13. The locations of the samples for tension bar and hardness and grain size measurements.

Hardness and grain size for T54 rings:

Hardness and grain size values are shown in Table 2 and tensile test results in table 3. Hardness variation is mainly 38-47 HV. In the grain size column, the results are presented for average values. The grain size variation for average values is 0,090-0,170 mm. Max values for single grains could be three times bigger in these samples. More microstructure/grain size data can be seen from Figures 14-16.

All values are below the specified hardness limit (<65 HV) and well below the maximum grain size limit (<0,360 mm).

Table 2. Hardness and grain size values for the T54 rings.

	Hardness [HV5]	**Grain size [mm]**	
		TOP	**BOTTOM**
TR 0°	42	0,100	0,100
TR 120°	41	0,120	0,100
TR 240°	47	0,090	0,100
CR 0°	39	0,120	-
CR 120°	39	0,170	-
CR 240°	38	0,150	-

BR 0°	40	0,120	-
BR 120°	40	0,120	-
BR 240°	40	0,120	-

Table 3. Tensile test results for T54.

	Yield Strength Rp0,2 [N/mm^2]	**Tensile Strength Rm [N/mm^2]**	**Elongation A5 [%]**
TR 0°	54	209	56
TR 120°	53	207	57
TR 240°	69	210	57
CR 0°	54	206	56
CR 120°	55	207	55
CR 240°	51	206	56
BR 0°	50	206	56
BR 120°	52	206	57
BR 240°	54	206	57

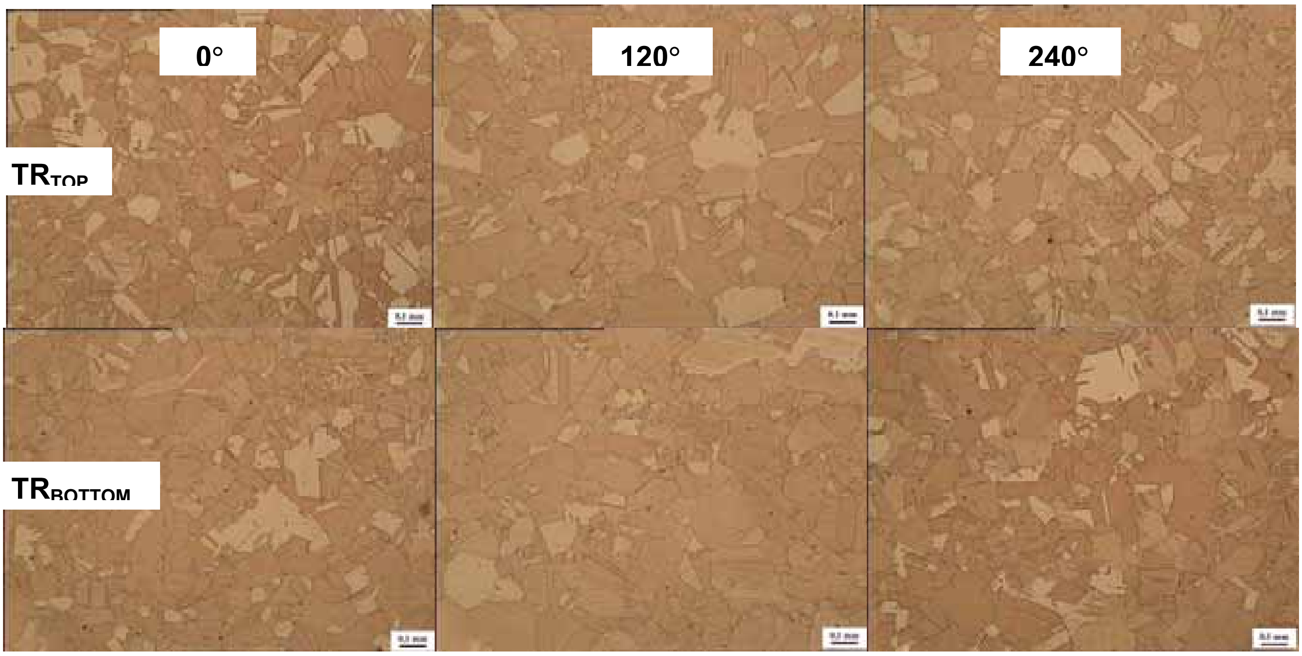


Figure14. T54 top ring microstructures at 0°, 120° and 240° circumferential position, at the top (upper row) and the bottom (lower row). 75x

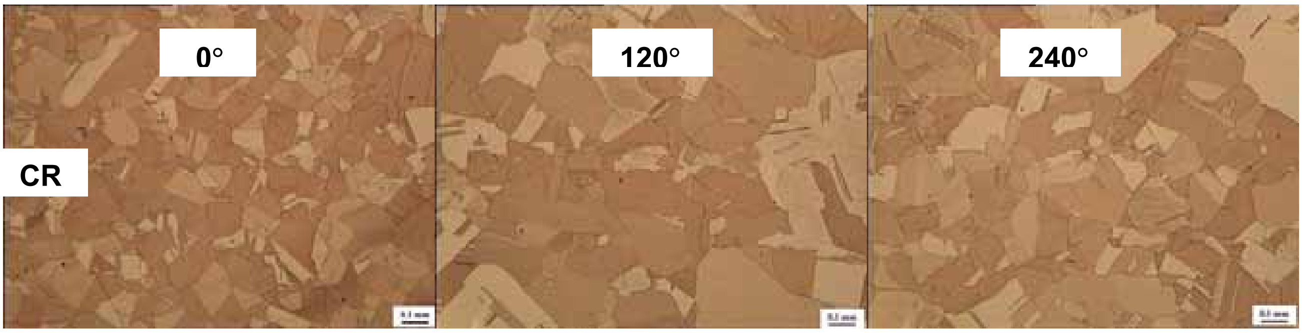


Figure 15. T54 centre ring microstructures at 0°, 120° and 240° circumferential position. 75x

Figure16. T54 base ring microstructures at 0°, 120° and 240° circumferential position. 75x

Some of the grains are not completely recrystallised. These areas can be found on almost all of the investigated samples (figures 14 to 16).

Wall thickness of T54 ring:

The tube T54 was investigated before machining. The measured thickness values differ significantly from the previous tubes, which had been machined. There is significant wall thickness variation on the top end of the tube (up to 13 mm). The wall thickness variation of center and bottom ends of the tube is less significant (2,9 mm and 1 mm). Measurements were carried out by calliper, which both measuring units were planar, so the actual values are a little bit lower level. All values are shown in Table 5.

Table 5. T54 Wall thickness for top ring (TR), centre ring (CR) and base ring (BR), at three circumferential locations 0°, 120° and 240°.

	TR [mm]	**CR [mm]**	**BR [mm]**
0°	72,50	78,85	114,0
120°	85,50	81,00	113,0
240°	81,20	81,70	113,0

CHEMICAL COMPOSITION

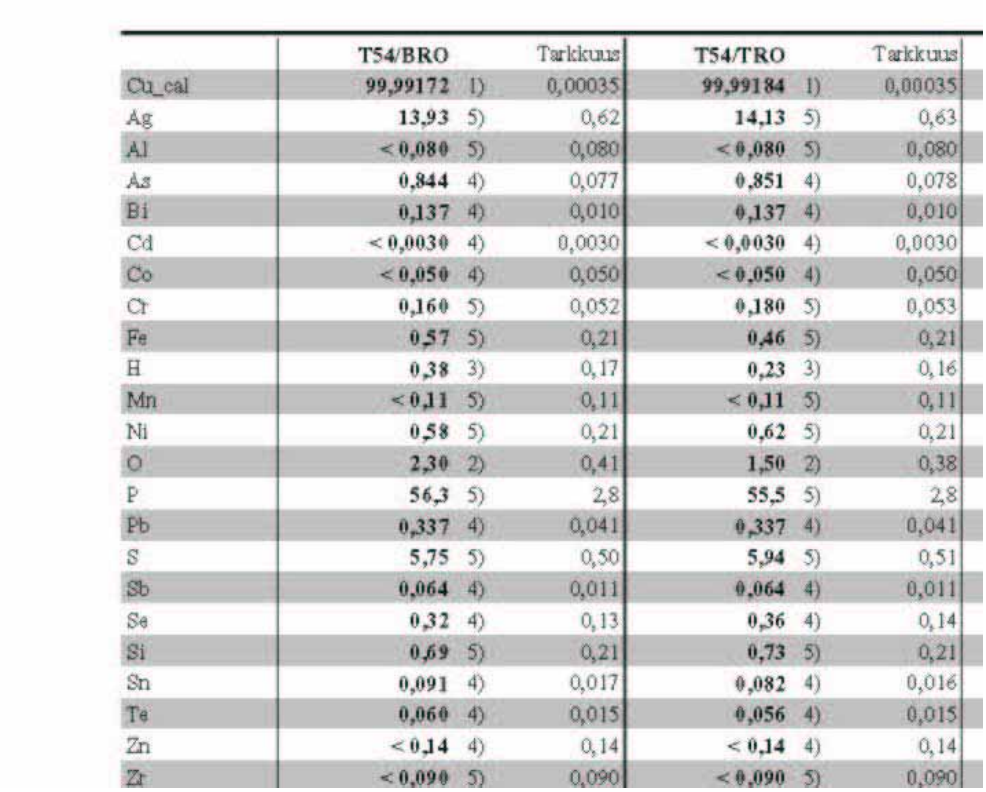
Chemical compositions were measured from two locations: top ring and base (0° -line). Measured values fulfil all requirements as can be seen from Table 6. More extensive analyses are given in Appendix 1.

Table 6. Chemical composition and requirements for the T54 top ring and the base (0° line).

	Requirement [ppm]	**TR 0° [ppm]**	**Base 0° [ppm]**
P	30-70	56	56
H	< 0,6	0,2	0,4
O	< 5	1,5	2,3
S	< 8	5,9	5,8

CONCLUSIONS

Nuclear canister tube T54 and tube bottom was cut to pieces and investigasted thoroughly at Luvata Pori Oy. There was significant improvement in grain size compared with the earlier results (T44 and T52). The grain sizes were clearly within the customer specification. However, some of the grains were not completely recrystallised. These areas could be found on almost all of the investigated samples. The improvements made on site (Mannesmann, June 2007) to the extrusion process clearly improved the properties of the tube and the bottom.



	T54/BRO		Tarkkuus	T54/TRO		Tarkkuus
Cu_cal	**99,99172**	1)	0,00035	**99,99184**	1)	0,00035
Ag	**13,93**	5)	0,62	**14,13**	5)	0,63
Al	**< 0,080**	5)	0,080	**< 0,080**	5)	0,080
As	**0,844**	4)	0,077	**0,851**	4)	0,078
Bi	**0,137**	4)	0,010	**0,137**	4)	0,010
Cd	**< 0,0030**	4)	0,0030	**< 0,0030**	4)	0,0030
Co	**< 0,050**	4)	0,050	**< 0,050**	4)	0,050
Cr	**0,160**	5)	0,052	**0,180**	5)	0,053
Fe	**0,57**	5)	0,21	**0,46**	5)	0,21
H	**0,38**	3)	0,17	**0,23**	3)	0,16
Mn	**< 0,11**	5)	0,11	**< 0,11**	5)	0,11
Ni	**0,58**	5)	0,21	**0,62**	5)	0,21
O	**2,30**	2)	0,41	**1,50**	2)	0,38
P	**56,3**	5)	2,8	**55,5**	5)	2,8
Pb	**0,337**	4)	0,041	**0,337**	4)	0,041
S	**5,75**	5)	0,50	**5,94**	5)	0,51
Sb	**0,064**	4)	0,011	**0,064**	4)	0,011
Se	**0,32**	4)	0,13	**0,36**	4)	0,14
Si	**0,69**	5)	0,21	**0,73**	5)	0,21
Sn	**0,091**	4)	0,017	**0,082**	4)	0,016
Te	**0,060**	4)	0,015	**0,056**	4)	0,015
Zn	**< 0,14**	4)	0,14	**< 0,14**	4)	0,14
Zr	**< 0,090**	5)	0,090	**< 0,090**	5)	0,090

Timo Välimäki / Research and development
Tuomas Renfors/ Metallurgical Applications

12.3.2008

81

Posiva

Testing of the base and the ring for the T55

12.3.2008

Distribution:

POSIVA: Veli-Matti Ämmälä, Tiina Jalonen

LUVATA PORI: Jouko Lammi, Timo Välimäki

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Sample preparation

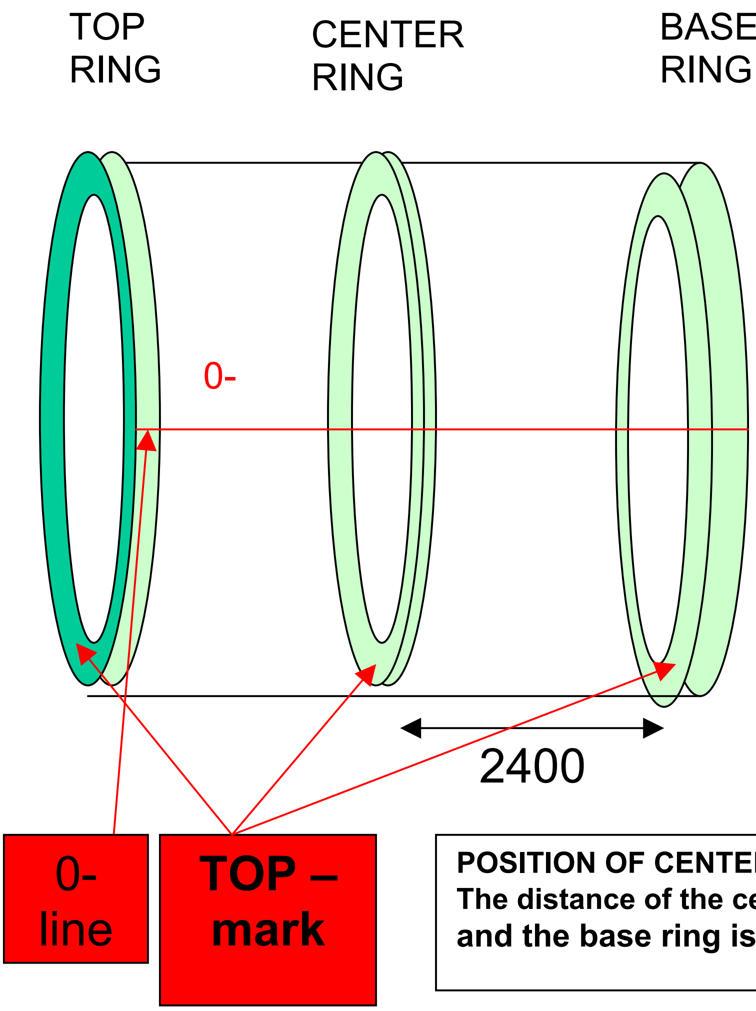
First the base was cut in half in two steps along the main diagonal (clockwise from 90° to 270°). Then the 25 mm slice was cut out from the base as we can see in Figure 1. After that, ¼ -slice (thickness 25 mm) was cut out from the base (clockwise from 180° to center) for more precise examinations.

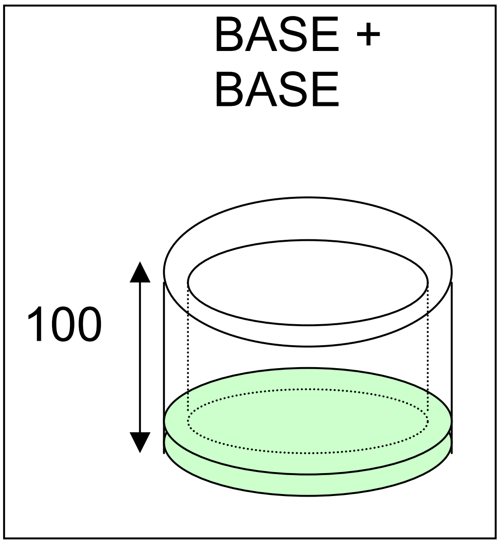
From the top ring, centre ring and the base ring 250 mm long sectors were cut out around the spots 0°, 120° and 240° (clockwise). Afterwards these sections and the base slices were examined in Luvata Pori according to separate instructions (in finnish).

T55 SAMPLING – Marks (on red)

TOP AND CENTER RING:
The width of the ring
(along the tube axis)
is 50 mm

BASE AND BASE RING:
The base will be cut off at
(50 mm base thickness + 50 mm
ring) = 100 mm
from the outer surface of the
base





POSITION OF CENTER RING:
The distance of the center ring
and the base ring is 2400 mm

Figure 1. Sample locations

INSPECTION OF THE TUBE BOTTOMS

T55 tube bottom

An etched macro slice from T55 base is shown in Figure 2. As figure 2 shows, the base is not machined to final dimensions. The outer diameter of the base is ~1085 mm, inner diameter ~925 mm and the thickness of the base is 163-185 mm. Schematic picture in Fig 3 shows coarse grain areas in the bottom. Grain structures are shown in figures 4 to 8. Coarse grained areas are visible on both sides of the centreline (Figs 5 and 7), while the grains at the centreline are rather small (Fig 6).

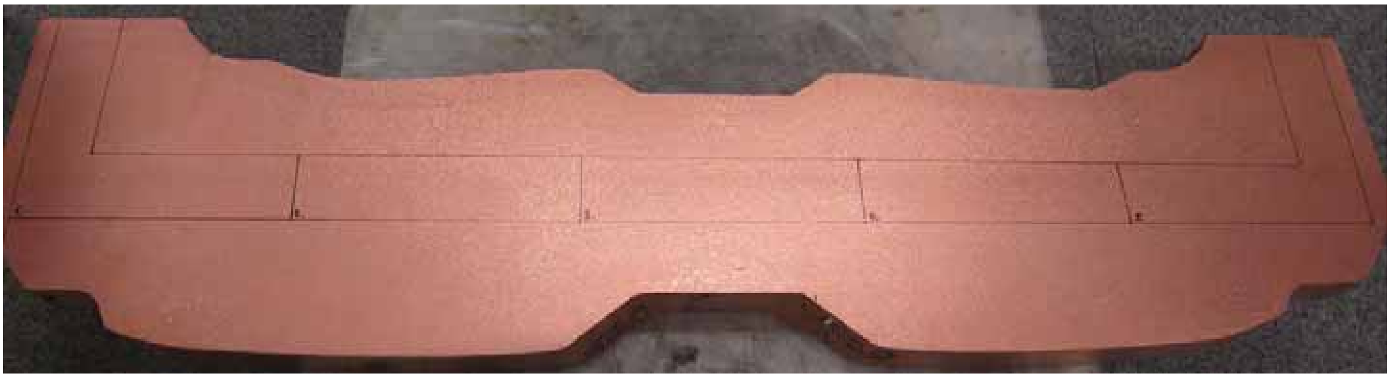


Figure 2 An etched base slice from T55 with sections drawn.

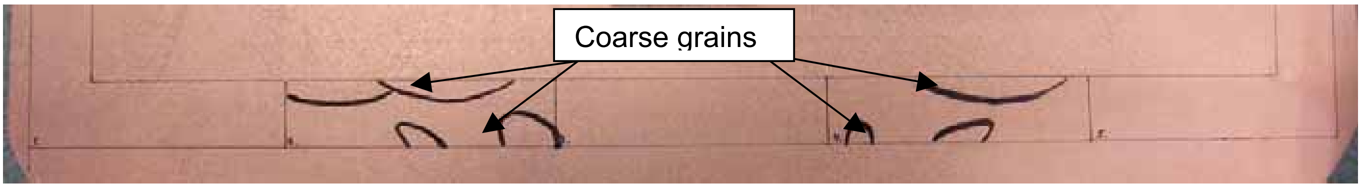


Figure 3. Coarse grain areas highlighted with black marker.

Figure 4. Corner area from the left hand side

Figure 5. Microstructure from the area right next to left corner.

Figure 6. **Center of the tube bottom.**

Figure 7 Microstructure from the area right next to right corner.

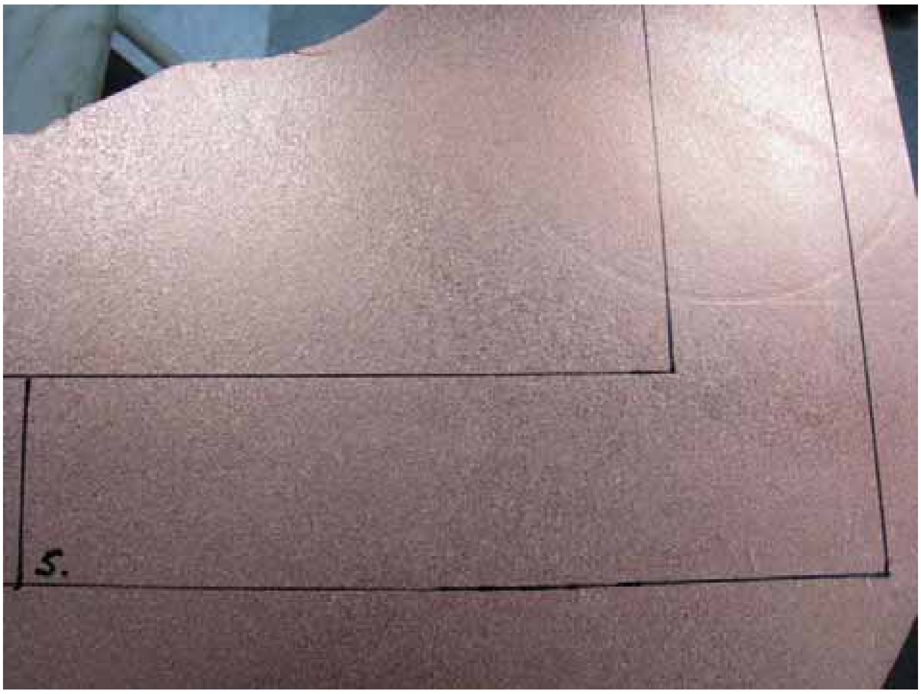


Figure 8. **Corner of the tube bottom on the right hand side.**

Hardness and grain size:

The ¼ -slice (thickness 25 mm) was cut transverse to the base slice. The ¼ -slice was cut into pieces as shown in Figure 9. Hardness and grain size examinations were made to both top and bottom side of the slide. Precise examination points are 140 (A), 210 (B), 290 (C), 370 (D) and 450 (E) mm from the outer diameter a.k.a in every 80 mm.

The grain size variation is 0,150-0,300 mm and results from the top and the bottom of the sample are close together. The grain size in the corner area is 0,210 mm. Grainsize varies toward the centreline (0,150-0,300 mm) and at the centreline the grainsize is at its smallest, 0,150-0,170 mm.

The hardness variation is 36-39 HV. The bottom is slightly softer in average than top. Hardness and grainsize results are shown in Table 1 and Figures 10 and 11. All samples are well below the specified hardness limit (<65 HV) and grain size limit (<0,360 mm).

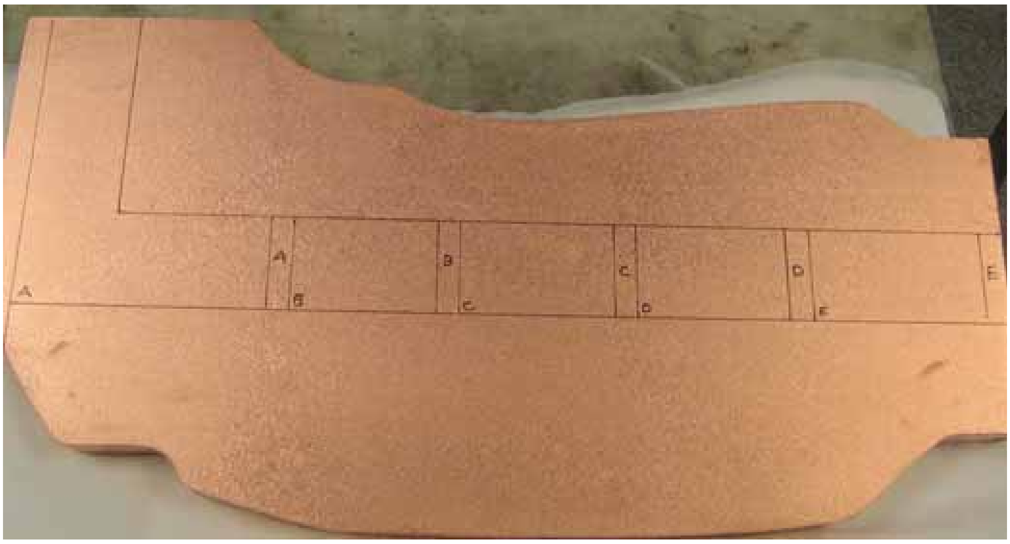


Figure 9 Sample locations for hardness and grain size measurements

Table 1. The hardness and grain sizes of T55 canister bottom along the inside radius every 80 mm towards to centre.

	Hardness [HV5]		**Grain size [mm]**	
	TOP	**BOTTOM**	**TOP**	**BOTTOM**
A	37	36	0,210	0,210
B	36	37	0,220	0,200
C	36	36	0,280-0.300	0,170
D	39	36	0,150	0,220
E	38	37	0,170	0,150

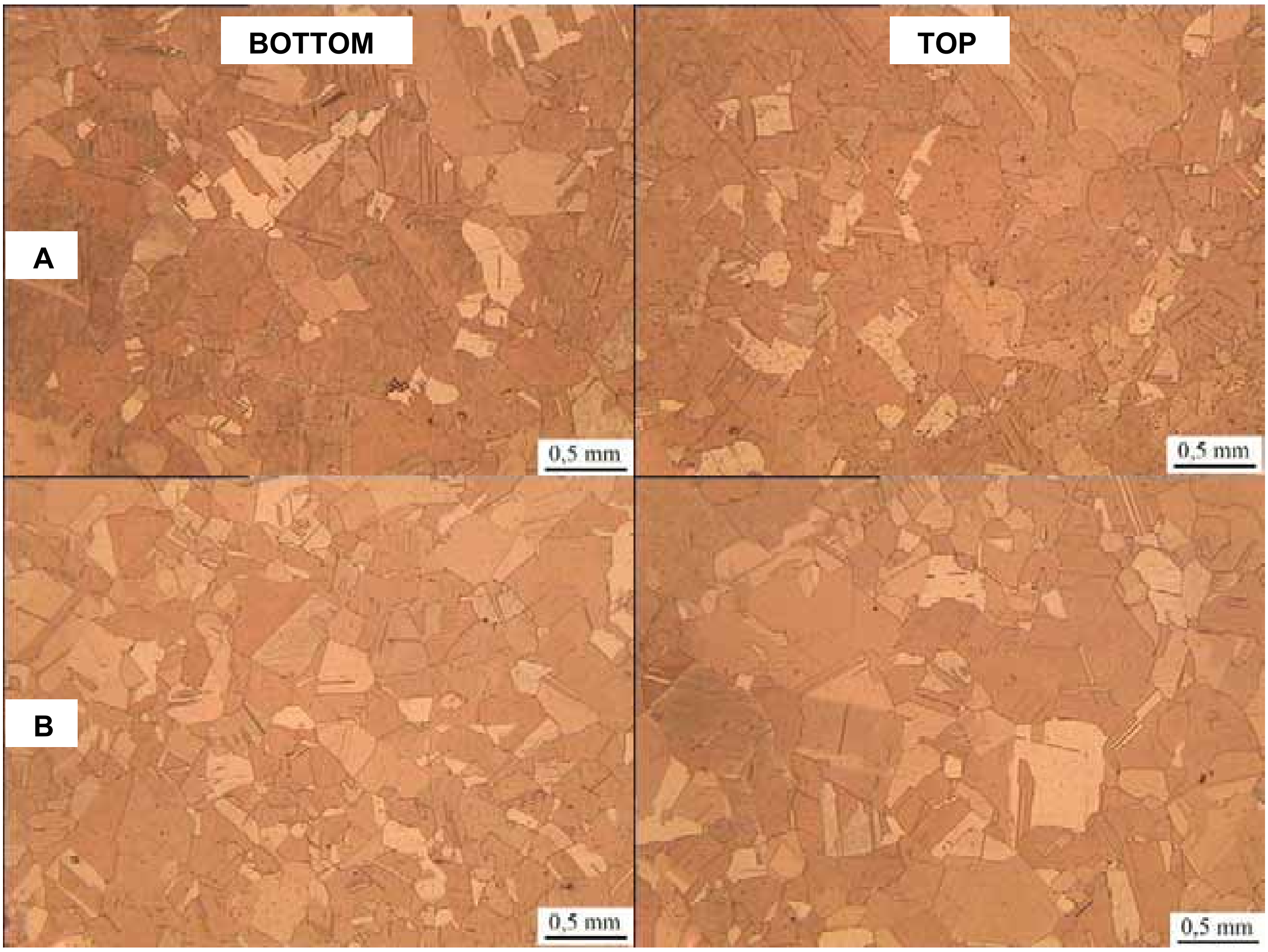


Figure 10 Microstructure from T55 sample A (upper - bottom/top) and B. 32x

Figure 11. **Microstructure from sample C (upper - bottom/top), D and E. 32x**

Some of the grains are not completely recrystallised. These areas can be found on almost all of the investigated samples (figures 10 and 11).

INSPECTION OF THE RINGS

The sectors for the examination were cut out from the rings at three circumferential locations 0°, 120° and 240° (Figure 12). The length of the sectors is about 250 mm and the rings are specified as Top Ring (TR), Center Ring (CR) and Base Ring (BR). Samples for tensile tests were taken from the inner circle of the ring. Samples for hardness and grain size measurements were taken from the outer circle of the ring. The locations of the samples are shown in Figure 13.

Figure 12. The sectors from the rings were cut out as above.

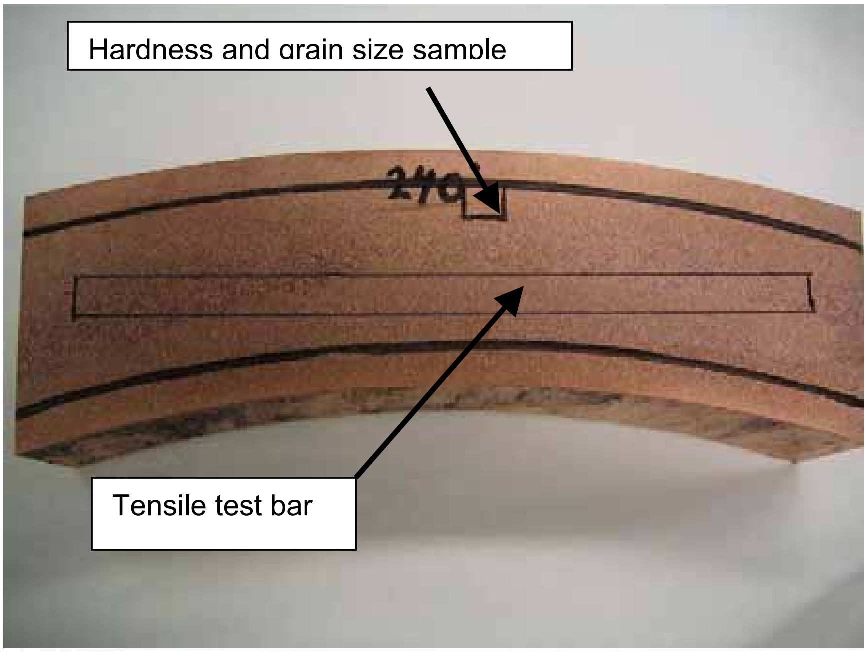


Figure13. The locations of the samples for tension bar and hardness and grain size measurements.

Hardness and grain size for T55 rings:

Hardness and grain size values are shown in Table 2 and tensile test results in table 3. Hardness is 37-40 HV. In the grain size column, the results are presented for average values. The grain size varies between 0,100 mm and 0,150 mm. Max values for single grains could be three times bigger in these samples. More microstructure/grain size data can be seen from Figures 14-16.

All values are below the specified hardness limit (<65 HV) and well below the maximum grain size limit (<0,360 mm).

Table 2. Hardness and grain size values for the T55 rings.

	Hardness [HV5]	**Grain size [mm]**	
		TOP	**BOTTOM**
TR 0°	38	0,120	0,120
TR 120°	39	0,120	0,100
TR 240°	40	0,120	0,120
CR 0°	39	0,140	-
CR 120°	37	0,150	-
CR 240°	39	0,120	-

BR 0°	38	0,150	-
BR 120°	40	0,120	-
BR 240°	39	0,120	-

Table 3. Tensile test results for T55.

	Yield Strength Rp0,2 [N/mm^2]	**Tensile Strength Rm [N/mm^2]**	**Elongation A5 [%]**
TR 0°	46	209	55
TR 120°	47	209	56
TR 240°	44	208	57
CR 0°	45	210	58
CR 120°	44	210	56
CR 240°	44	208	55
BR 0°	46	208	55
BR 120°	45	207	55
BR 240°	47	207	55

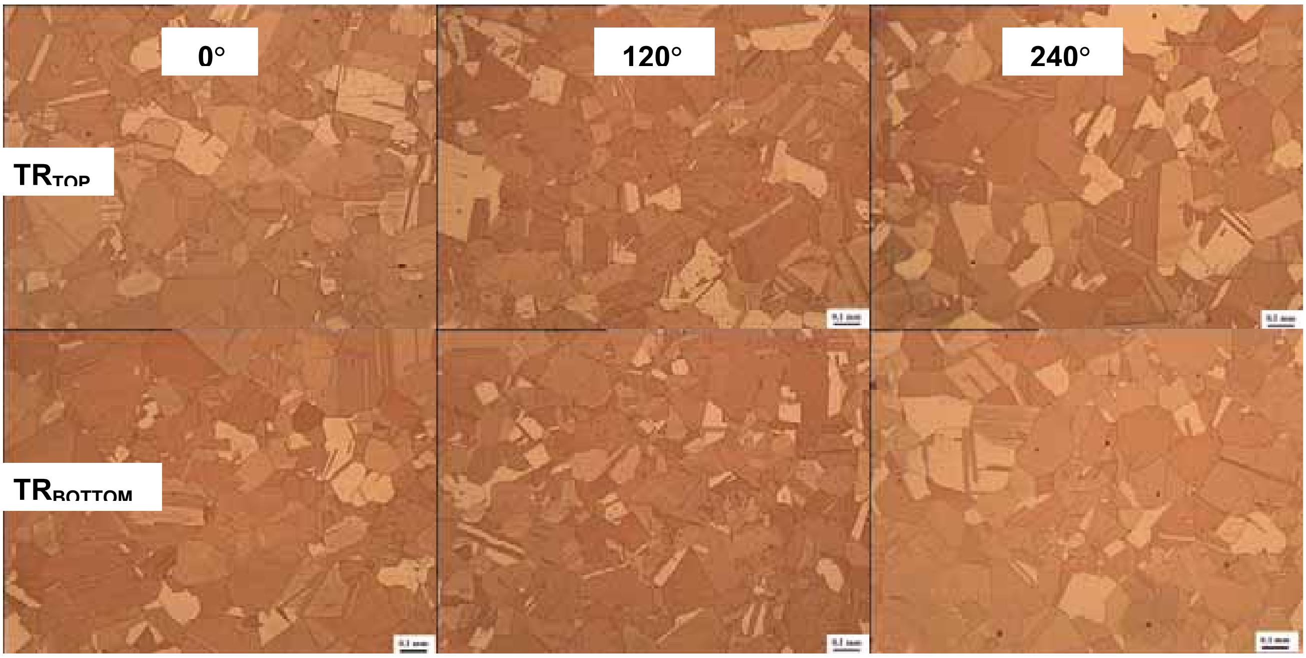


Figure14. T55 top ring microstructures at 0°, 120° and 240° circumferential position, at the top (upper row) and the bottom (lower row). 75x

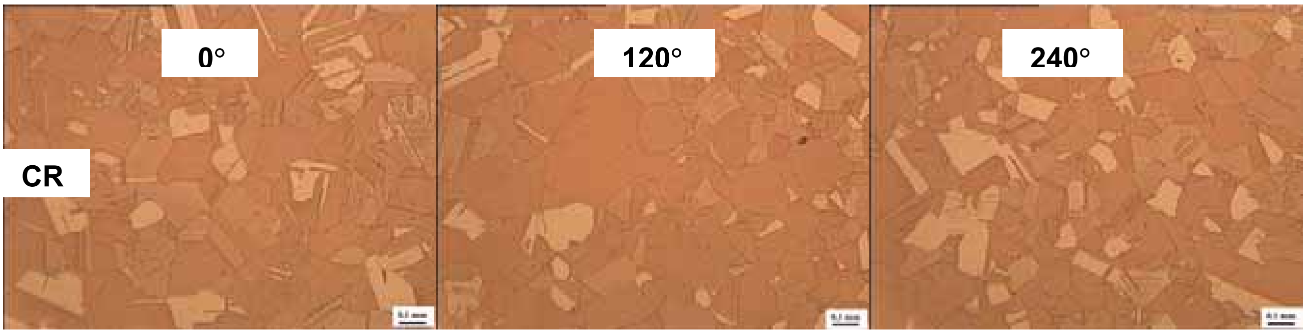


Figure 15. T55 centre ring microstructures at 0°, 120° and 240° circumferential position. 75x

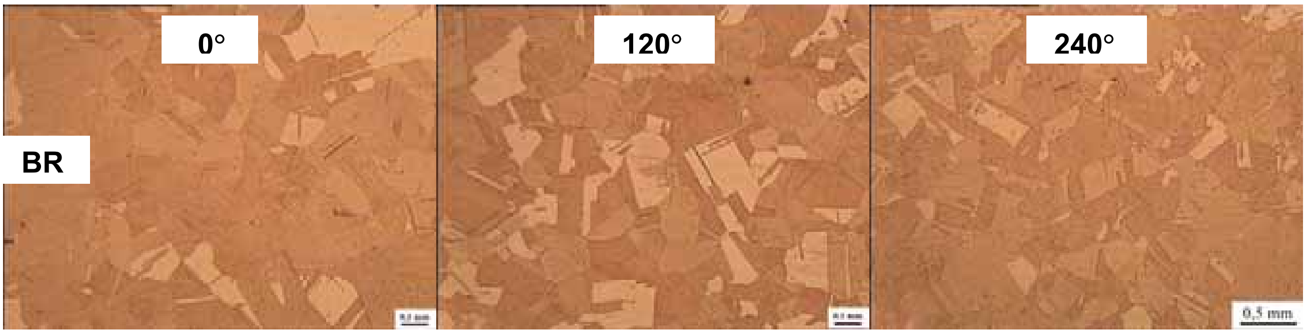


Figure16. T55 base ring microstructures at 0°, 120° and 240° circumferential position. 75x

The recrystallisation of grain structure is almost complete. Improvement in grain structure homogeneity is significant compared with the previous tube T54.

Wall thickness of T55 ring:

The tube T55 was investigated before machining. The wall thickness variation of the tube was very small. Measurements were carried out by calliper, which both measuring units were planar, so the actual values are a little bit lower level. All values are shown in Table 5.

Table 5. T55 Wall thickness for top ring (TR), centre ring (CR) and base ring (BR), at three circumferential locations 0°, 120° and 240°.

	TR [mm]	**CR [mm]**	**BR [mm]**
0°	72,0-76.0	75.0-76.5	108,0
120°	74.5-76.5	75.3-75.9	121,0
240°	76.5-79.0	76.5-77.0	113,0

CHEMICAL COMPOSITION

Chemical compositions were measured from two locations: top ring and base (0° -line). Measured values fulfil all requirements as can be seen from Table 6. More extensive analyses are given in Appendix 1.

Table 6. Chemical composition and requirements for the T55 top ring and the base (0° line).

	Requirement [ppm]	**TR 0° [ppm]**	**Base 0° [ppm]**
P	30-70	50	53
H	< 0,6	0,21	0,22
O	< 5	1,0	1,9
S	< 8	5,71	5,74

CONCLUSIONS

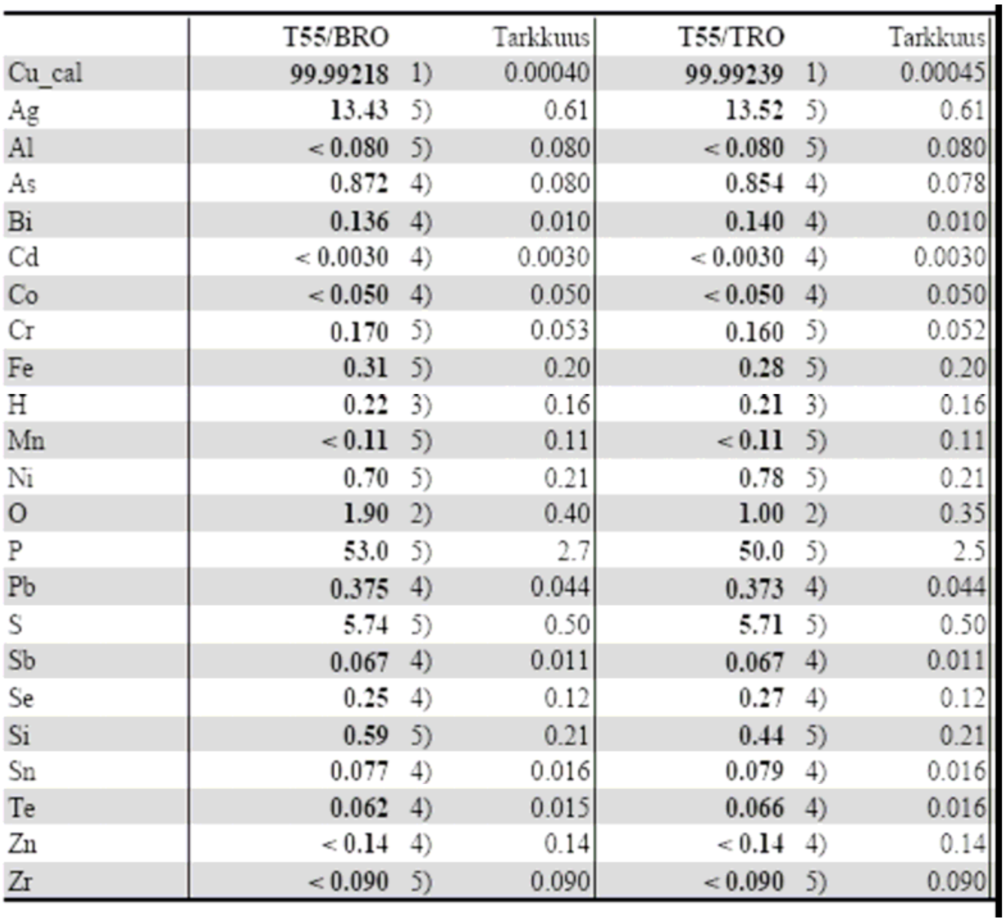
Nuclear canister tube T55 and tube bottom were cut to pieces and investigated thoroughly at Luvata Pori Oy. There was significant improvement in the grain structure of the tube wall compared with the earlier results (T54). The improvement was achieved by adjusting the process parameters at Mannesmann. The process adjustments were made to the piercing and final forging temperatures. The piercing temperature however was lower than anticipated due to the problems in lifting the blank.

The grain structure of CR-ring was almost optimal. The recrystallisation degree was high and grain size variation was minimal. TR-ring had some larger grain regions. However, the spec limit for grain size was not exceeded. The grain size variation problem of the upper part of the ring can be solved by cutting more scrap from the top end.

The grain structure of bottom of the tube contained as-cast grain regions. The grain structure of the bottom of the tube is formed mainly during the piercing operation. Later operations have only minor effect on the structure of the bottom.

Vallourec&Mannesmann measurements of tubes T54 and T55 shows, that there is difference in bottom thicknesses after piercing operation. The piercing depth of tube T54 was greater than of tube T55. For future experiments it is important to acknowledge the importance of adequate lubrication during the piercing operation and also the exact piercing depth, since it has major effect on the final grain structure of the bottom. One possible way to further deform the bottom of the tube is to pierce it further to make the bottom thinner. Thinner bottom deforms easier during the following processing steps as inner diameter of the tube is increased.

The latest improvements made on site (Mannesmann, December 2007) to the extrusion process improved particularly the properties of the tube wall.



	T55/BRO		Tarkkuus	T55/TRO		Tarkkuus
Cu_cal	99.99218	1)	0.00040	99.99239	1)	0.00045
Ag	13.43	5)	0.61	13.52	5)	0.61
Al	< 0.080	5)	0.080	< 0.080	5)	0.080
As	0.872	4)	0.080	0.854	4)	0.078
Bi	0.136	4)	0.010	0.140	4)	0.010
Cd	< 0.0030	4)	0.0030	< 0.0030	4)	0.0030
Co	< 0.050	4)	0.050	< 0.050	4)	0.050
Cr	0.170	5)	0.053	0.160	5)	0.052
Fe	0.31	5)	0.20	0.28	5)	0.20
H	0.22	3)	0.16	0.21	3)	0.16
Mn	< 0.11	5)	0.11	< 0.11	5)	0.11
Ni	0.70	5)	0.21	0.78	5)	0.21
O	1.90	2)	0.40	1.00	2)	0.35
P	53.0	5)	2.7	50.0	5)	2.5
Pb	0.375	4)	0.044	0.373	4)	0.044
S	5.74	5)	0.50	5.71	5)	0.50
Sb	0.067	4)	0.011	0.067	4)	0.011
Se	0.25	4)	0.12	0.27	4)	0.12
Si	0.59	5)	0.21	0.44	5)	0.21
Sn	0.077	4)	0.016	0.079	4)	0.016
Te	0.062	4)	0.015	0.066	4)	0.016
Zn	< 0.14	4)	0.14	< 0.14	4)	0.14
Zr	< 0.090	5)	0.090	< 0.090	5)	0.090