RESEARCH REPORT

Creep of copper with different NDT sound attenuation

Karin Mannesson, Henrik C.M. Andersson-Östling

KIMAB-2013-124
Open

Contract Research

swerea | KIMAB

Swerea KIMAB AB ● Box 7047 SE-164 07 Kista, Sweden
Tel. +46 (0)8 440 48 00 ● Fax +46 (0)8 440 45 35 ● kimab@swerea.se ● www.swereakimab.se

Creep of copper with different NDT sound attenuation

Authors: Karin Mannesson, Henrik C.M. Andersson-Östling
Report No: KIMAB-2013-124
ISSN: 1403-848X
Print: Printshop, city and year

swerea KIMAB

Creep of copper with different NDT sound attenuation

Author: Karin Mannesson, Henrik C.M. Andersson-Östling

Report No.: KIMAB-2013-124
Date: 2013-10-08
Member programme: Contract research
Swerea KIMAB Project No.: 26 40 17-18
Status: Open

Abstract

Material taken from three copper tubes intended for nuclear waste disposal has been creep tested at 125 °C. Two tubes had been manufactured by extrusion and one tube by the pierce-and-draw method. All tubes had been selected for testing since they had exhibited higher than normal sound attenuation during non-destructive ultrasound testing. The results from the creep testing show that no significant difference in creep response exists for the three tubes. Norton stress exponents and ductility values are also within normal limits for similar copper testing. The microstructure was also studied and the extruded tubes showed a grain structure consisting of a mixture of small and large grains. The pierced-and-drawn tube has almost only large grains.

Swerea KIMAB AB ●Box 7047, SE-164 07 Kista, Sweden
Tel. +46 (0)8 440 48 00 ● Fax +46 (0)8-440 45 35 ● kimab@swerea.se ● www.swereakimab.se

Table of Contents

1. Background

Nuclear waste in Sweden is planned to be disposed of by encapsulating in copper canisters and buried deep into the bedrock. The actual fuel elements will be inserted in a cast iron canister, which in turn will be inserted in a copper shell. The cast iron insert is bearing the load from the outer hydrostatic pressure and the copper shell is providing corrosion protection.

The canisters will be 5 m in length, have a 1 m diameter and have copper shell thickness of 5 cm. An overview of the design is given in [1]. When manufacturing the copper shells two different methods has been studied, extrusion of tubes or pierced-and-drawn tubes. Both methods give copper tubes that fulfil dimensional criteria. After manufacturing, the produced raw tubes are subjected to non-destructive testing (NDT) such as ultrasound inspection. In the NDT step some areas of the manufactured tubes exhibit higher sound attenuation than normal, [1, page 78-80]. The high attenuation is due to some difference in the copper in just that area, likely a different grain size from normal. A parallel project has studied the levels of oxygen, phosphorous and sulphur in the high attenuation copper to evaluate if mass segregation or depletion of alloying elements takes place [2].

The aim of the present work is to study the creep response of copper taken from areas of different sound attenuation. The work will also provide insights on the acceptable variation of grain size on the copper.

2. Materials and experimental

2.1 Materials

In this study material from three different tubes is tested. All materials are phosphorus doped, oxygen free copper (Cu-OFP). The material is taken from the extruded tubes T53 and T58 and the pierced-and-drawn tube T64. Five specimens were manufactured from each tube. The specimens were cut out from special zones (named 114-190, 200-240 and 155-185, corresponding to the angle of rotation from the zero-rotation marking), Figure 1. The parts of the tubes are assumed to be homogenous within the received parts and they clearly showed high sound attenuation compared with other parts of the tube. Small pieces were also cut out for measurements of hardness and chemical composition.

Figure 1. The three different tube pars received for testing. The markings show the different positions for the creep specimen blanks.

The creep specimens are of plain type with quadratic cross section 9*9 mm, 80 mm in gauge length and 153 mm in total length, Figure 2. To prevent cold deformation the specimens were electric discharged machined (EDM) and no annealing was applied either before or after the manufacturing step.

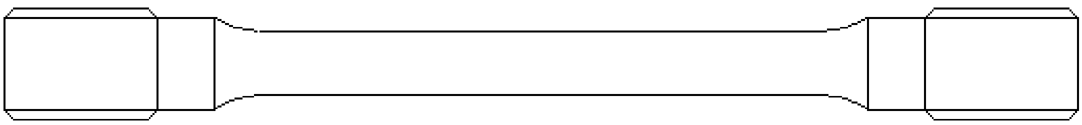

Figure 2. Specimen drawing.

2.2 Experimental

Iso-thermal creep tests were performed at 125° C. For each series five tests were done and the stress was chosen to give rupture times between 500 and 3000 hours. Previous experience with using dead-weight lever creep test rigs has shown that the repeated unloading connected with the reset of the lever arm after each 5 mm creep elongation yield problems with the total result. New test rigs with a maximum travel of 80 mm were therefore developed, and these rigs have been used for the testing in this project. The test rigs are further described in [3].

Due to the way the test machines work the test is stopped some 30 seconds before total separation of the specimen. The specimen is thus still in one piece after testing.

Hardness measurements and chemical composition was studied in another project connected to this, [2], and for completeness the results will be given in the results section.

3. Results

3.1 Creep testing

The results from the creep testing can be found in Table 2. Figure 3 shows stress versus creep rupture time in a log-log plot with trend lines added for all series. Similar creep behaviour is seen for all three tubes where the rupture time increases with decreasing stress. Since no previous results for material with normal sound attenuation tested with the same type of specimen manufacture is available, no comparison has been made. Further work is needed to develop the understanding more fully.

The minimum creep rate versus stress is plotted in Figure 4 showing a linear relation between creep rate and stress following the Norton power law, Eqn. 1 [4]. The calculated Norton exponents are listed in Table 1. The pierced-and-drawn tube T64 has a much higher exponent than the extruded tubes T53 and T58; this difference is also seen in the Norton plot in Figure 4.

Eqn 1. $$\dot{\varepsilon} = A_0 \exp(-\frac{Q}{RT})\sigma^n$$

Where ε is the creep rate, A_0 is a material constant, *Q* the activation energy for creep, *T* the temperature, *R* the gas constant, σ the stress and *n* the Norton exponent.

Table 1 Norton stress exponents.

Material	Norton exponent
T64	67
T58	20
T53	30

Table 2 Test matrix and creep results.

Test ID	Temperature [°C]	Stress* [MPa]	Loading time [h]	Creep Time [h]	Total Time [h]	Loading strain** [%]	Creep strain** [%]	Total strain** [%]	Area red [%]	Min creep rate [%/h]
T64 195	125	209		0			0.0			
T64 175	125	155	0.37	13.3	13.6	21.5	21.2	42.7	48.6	0.72
T64 165	125	145	0.33	463	463	24.0	27.7	51.7	58.0	0.02820
T64 155	125	150	0.27	111	111	23.7	30.9	54.6	55.7	0.1216
T64 185	125	140	0.33	858	858	23.8	30.5	54.2	59.4	0.00120
T58 240	125	160	0.35	69	69	23.2	16.3	39.5	74.7	0.1374
T58 230	125	155	0.23	267	267	22.2	26.7	48.9	87.3	0.031499
T58 220	125	145	0.33	1018	1018	16.7	28.6	45.3	59.8	0.00904
T58 210	125	150	0.35	288	288	17.6	32.2	49.8	55.3	0.07022
T58 200	125	140	0.35	581	581	17.1	34.7	51.8	65.0	0.00860
T53 190	125	160	0.33	14.8	15.1	23.8	16.4	40.2	80.8	0.589
T53 180	125	155	0.28	95	95	22.8	22.3	45.1	52.0	0.10548
T53 170	125	145	0.29	448	448	20.3	34.6	54.9	54.0	0.0519
T53 124	125	150	0.39	238	239	18.0	28.3	46.3	56.2	0.026667
T53 114	125	140	0.24	1445	1445	14.9	31.8	46.7	59.5	0.00541

* Nominal stress at the start of the test
** Technical strain measurements

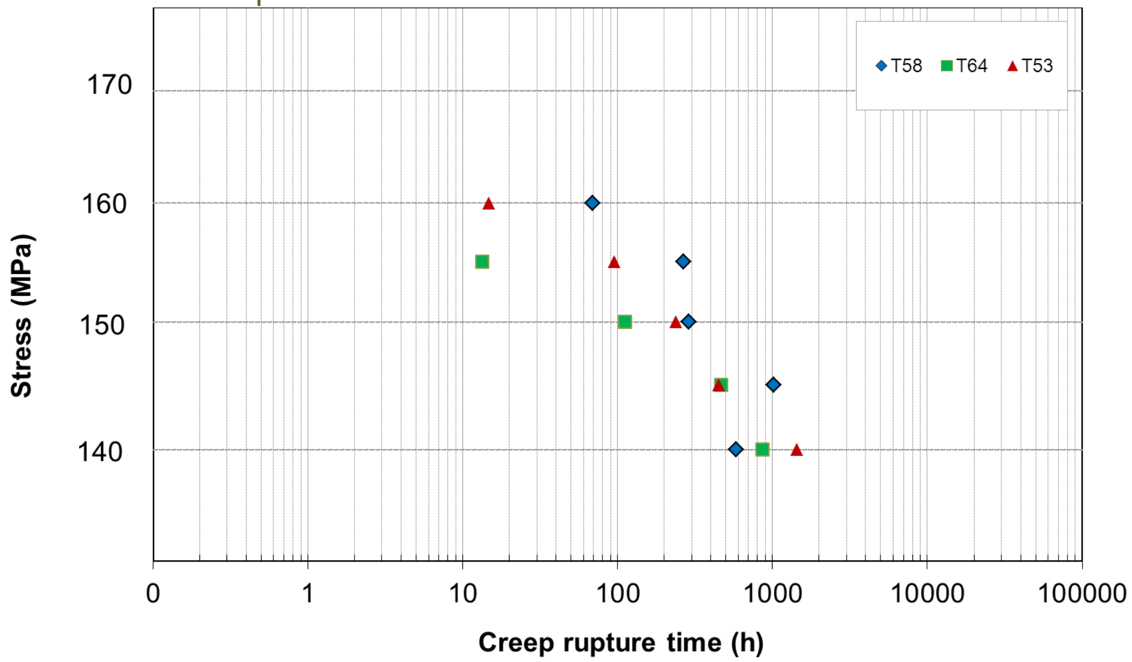

Figure 3. Applied stress against rupture time for all creep specimens.

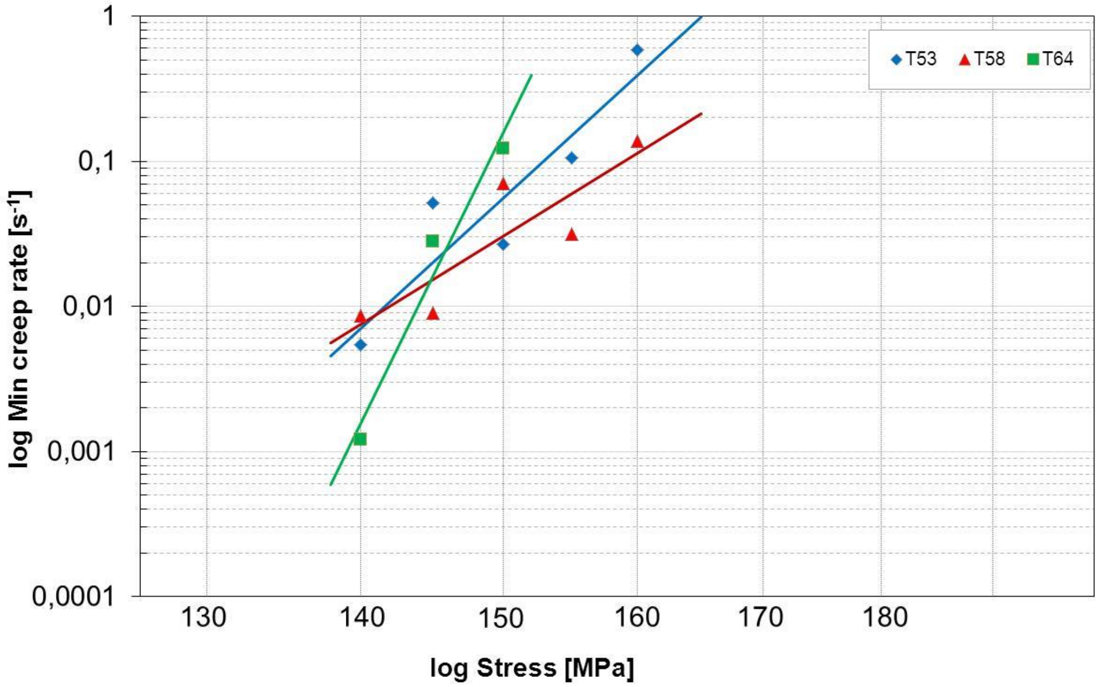

Figure 4. Minimum creep rate against applied stress for all specimens.

Figure 5, Figure 6 and Figure 7 show the creep ductility in form of loading strain, creep strain and total strain versus stress, respectively. The loading strain shows a slightly higher strain for the T64 tube. The creep strain shows more scatter than the loading strain and all creep strains lie over 15%. When looking at the total strain all specimens lie between 39% and 55%. No clear difference between the extruded and pierced-and-drawn tube is seen in the strain behaviour. When looking at the area reduction, Figure 8, a scatter between 48% and 87% is seen. Three specimens, T58-230, T58-240 and T53-190 show a much higher area reduction.

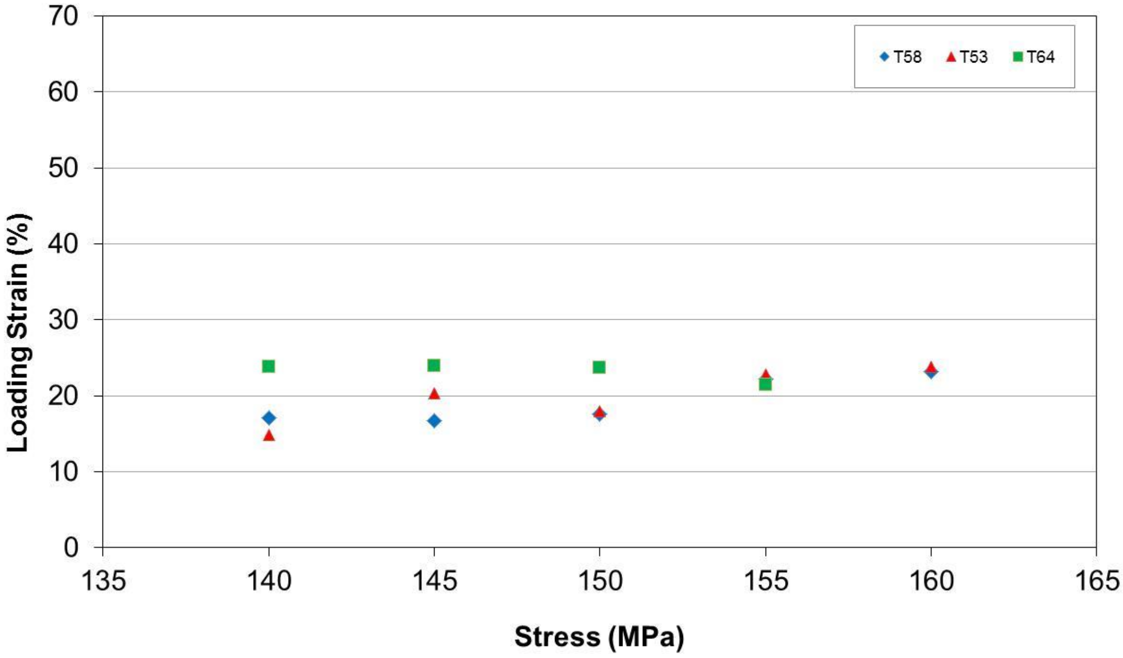

Figure 5. Loading strain against applied stress for all specimens.

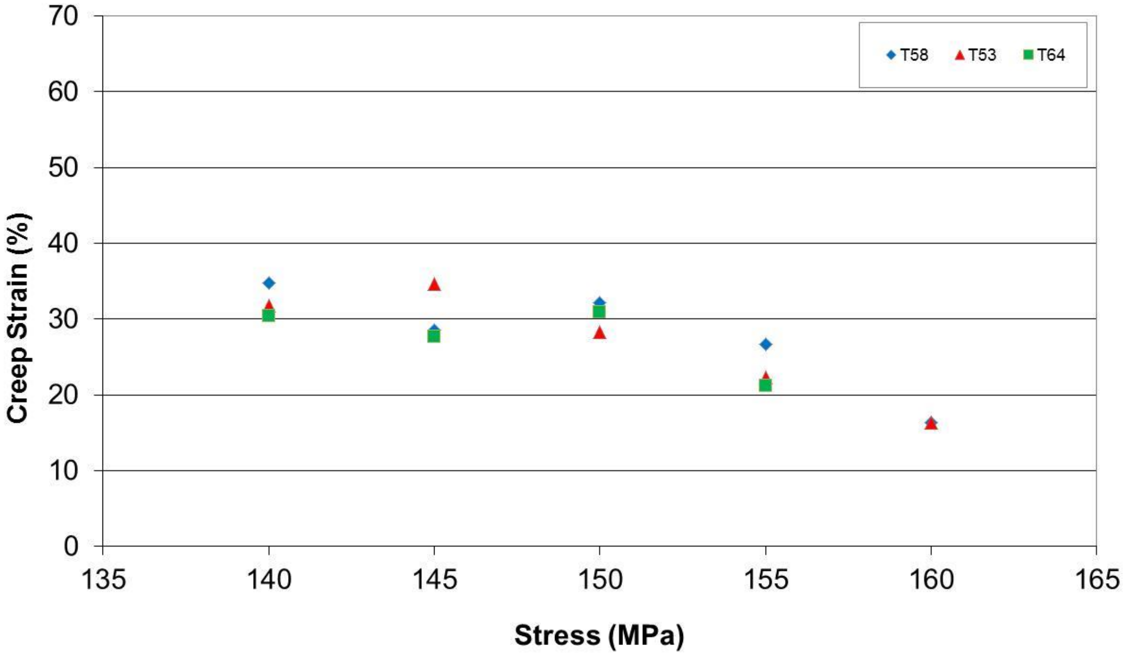

Figure 6. Creep strain against applied stress for all specimens.

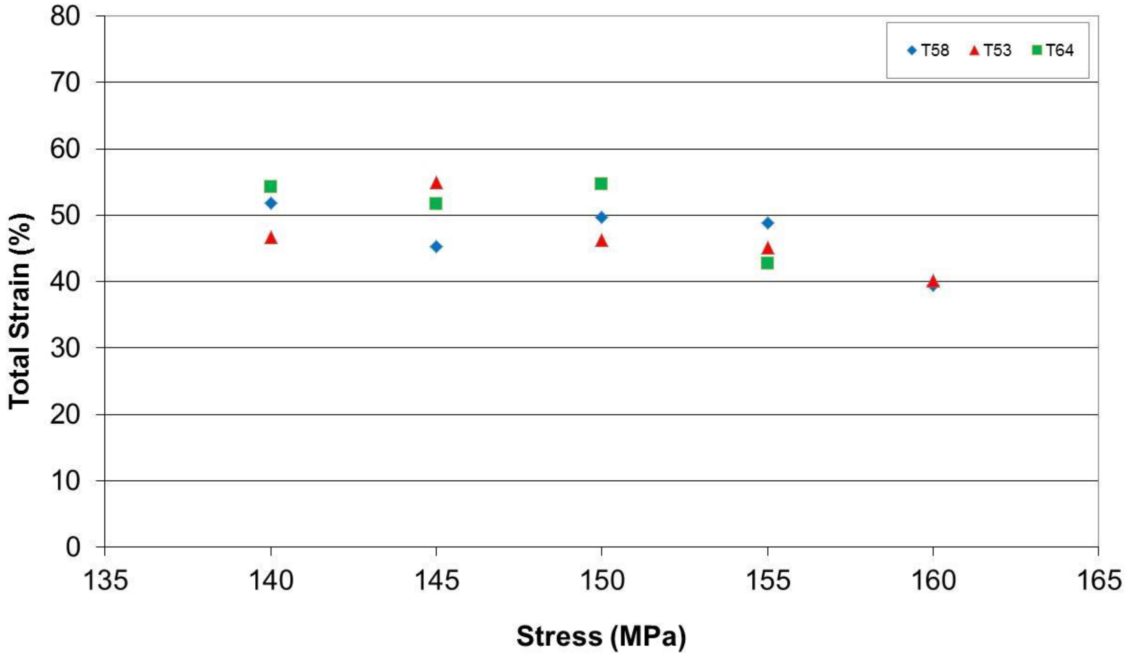

Figure 7. Total strain against applied stress for all specimens.

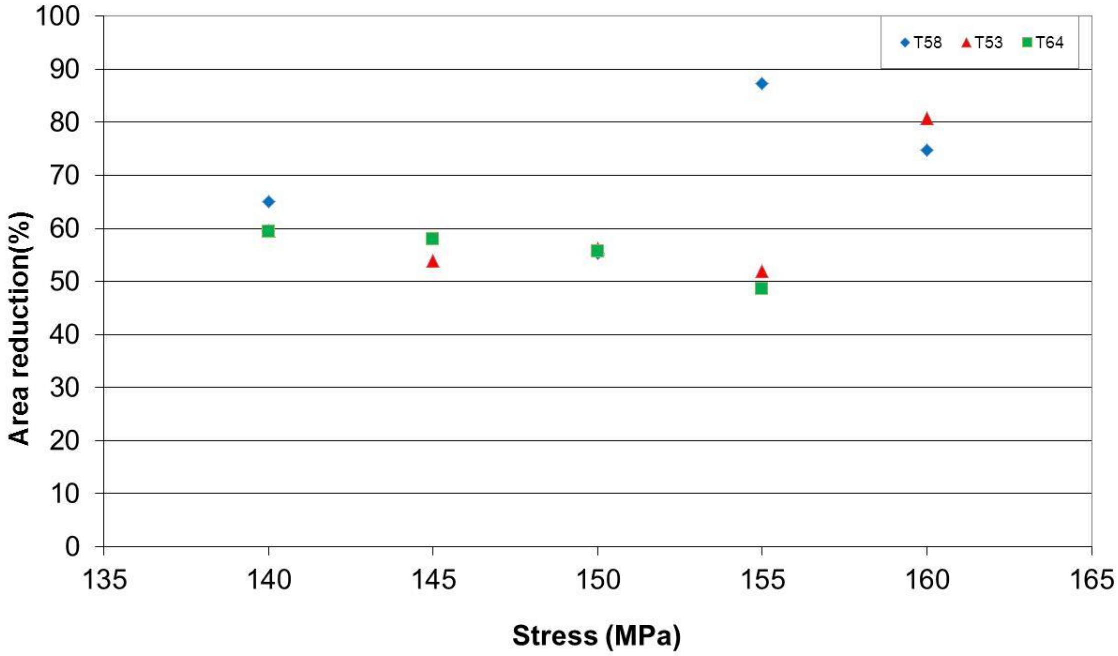

Figure 8. Area reduction against applied stress for all specimens.

3.2 Metallography

Images of the microstructure in the tested materials are given in Figure 10 through Figure 19. The structure in the extruded tubes T53 and T58 is more complex than in the pierced-and-drawn tube T64. This since the microstructure is made up from both large and small grains in a random pattern. By contrast tube T64 shows a structure consisting of almost totally larger grains. Twins are abundant but have not been taken into account for the grain size estimation. The same amount of twinning as in T53 and T58 is present in T64.

Grain size measurements were done on three positions on the different tubes, near the outer surface (A), in the middle (B) and near the inner surface (C), Figure 9 and Table 3. The grain size was also studied on material from near the fracture of the specimens. The specimens were mounted, ground and polished, finally etched with a solution for copper consisting of 40 g CrO_3, 7.5 g NH_4Cl, 50 ml H_2SO_4, 50 ml HNO_3 and 1900 ml H_2O. Grain size was measured using Jeffries method [5]. The grain size is noticeably smaller near the outer surface, and increasing further inward towards the inner surface. Note that the average grain size does not reflect the occurrence of large and small grains in the same structure as mentioned above.

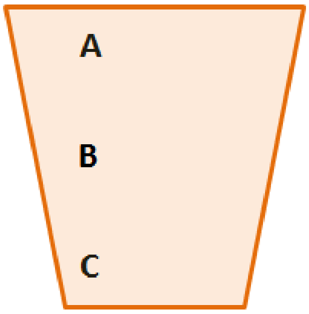

Figure 9. Picture showing the positions for grain size measurements.

Table 3 Grain size measurements in the three tubes T53, T58 and T64.

Material	**Grain size [µm]**		
	A	**B**	**C**
T64	96	174	211
T58	128	189	198
T53	135	242	235

After testing the microstructure takes on an elongated appearance, Figure 19. Observations during the microscopy work indicate that the larger grains are less deformed than the small grains, but no qualitative evidence could be found during the present studies. More microscopy work and possibly modelling of the creep deformations in a complex structure is needed to fully understand the creep process on the grain size level.

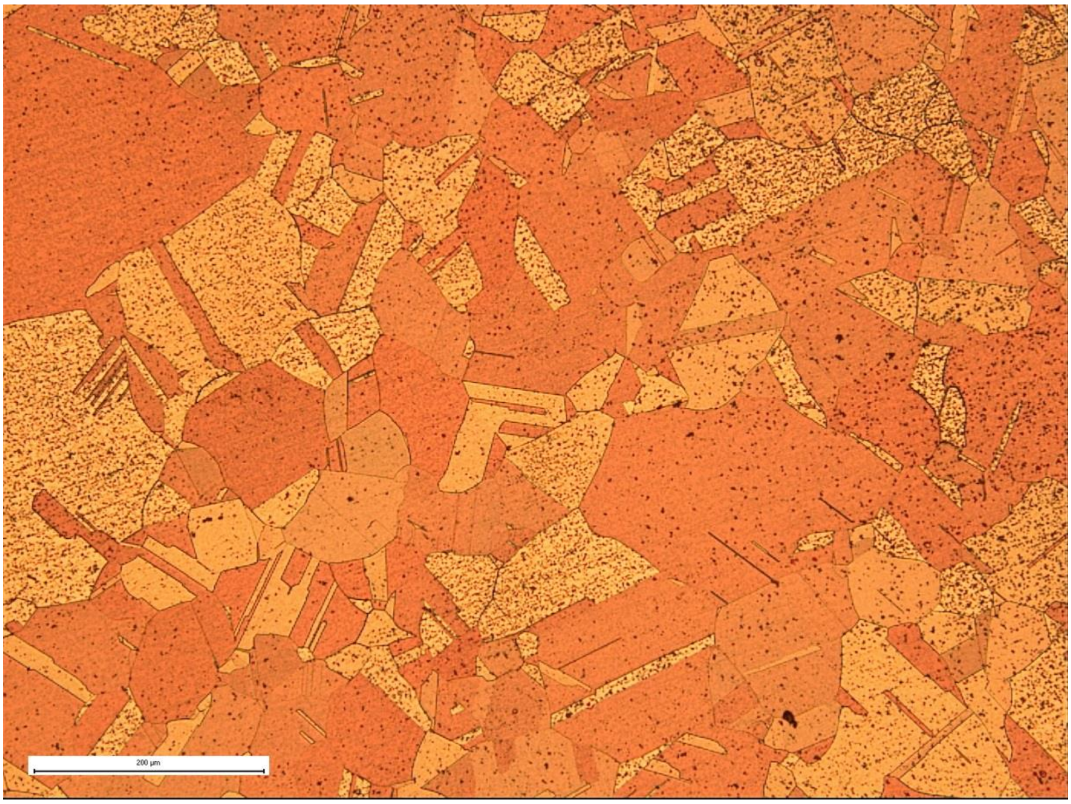

Figure 10. T53: Area A 100x.

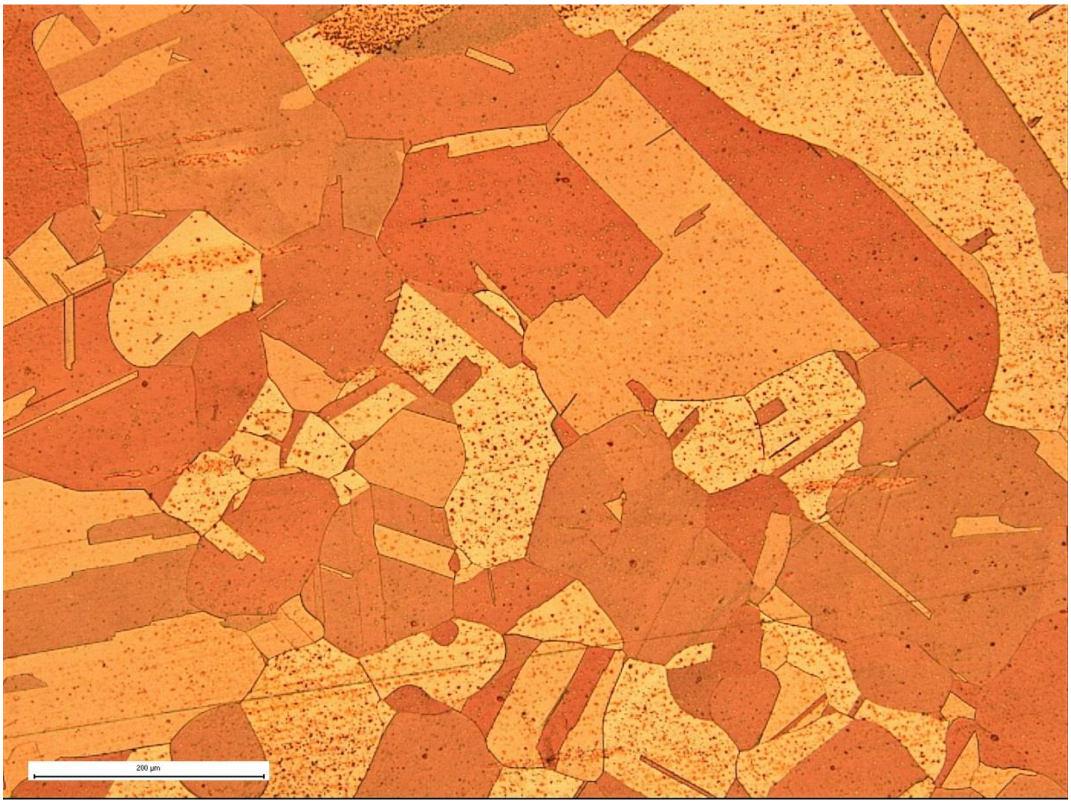

Figure 11. T53: Area B 100x.

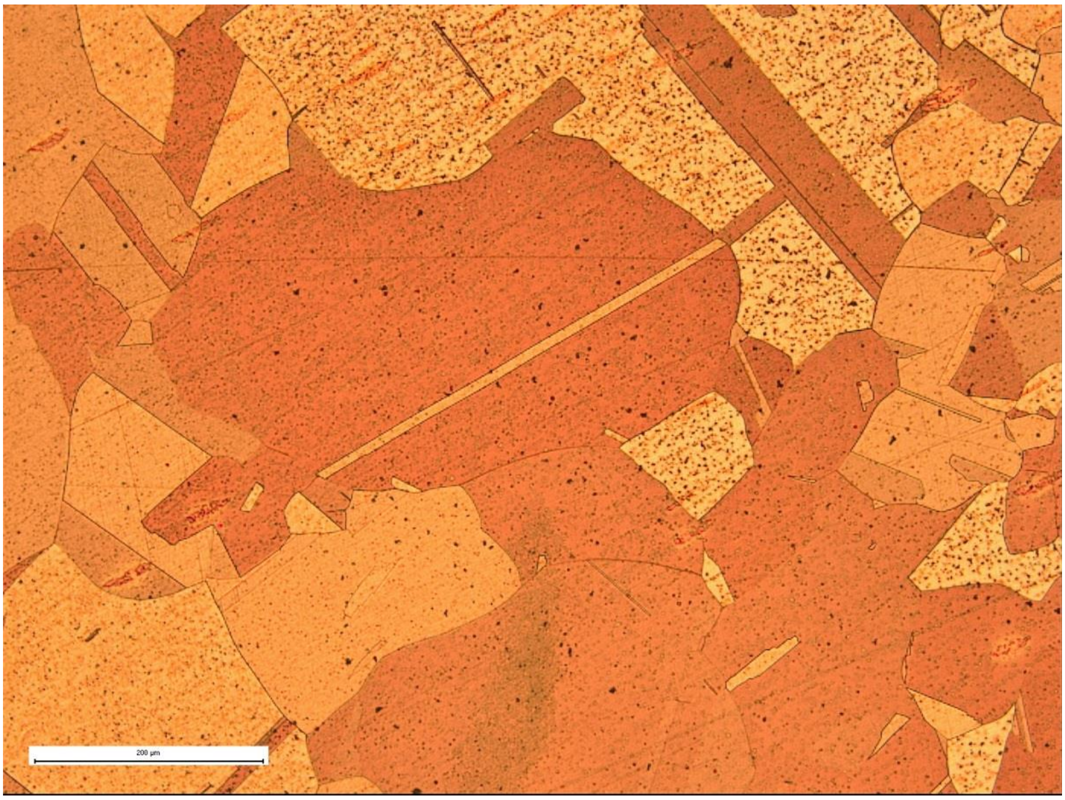

Figure 12. T53: Area C 100x.

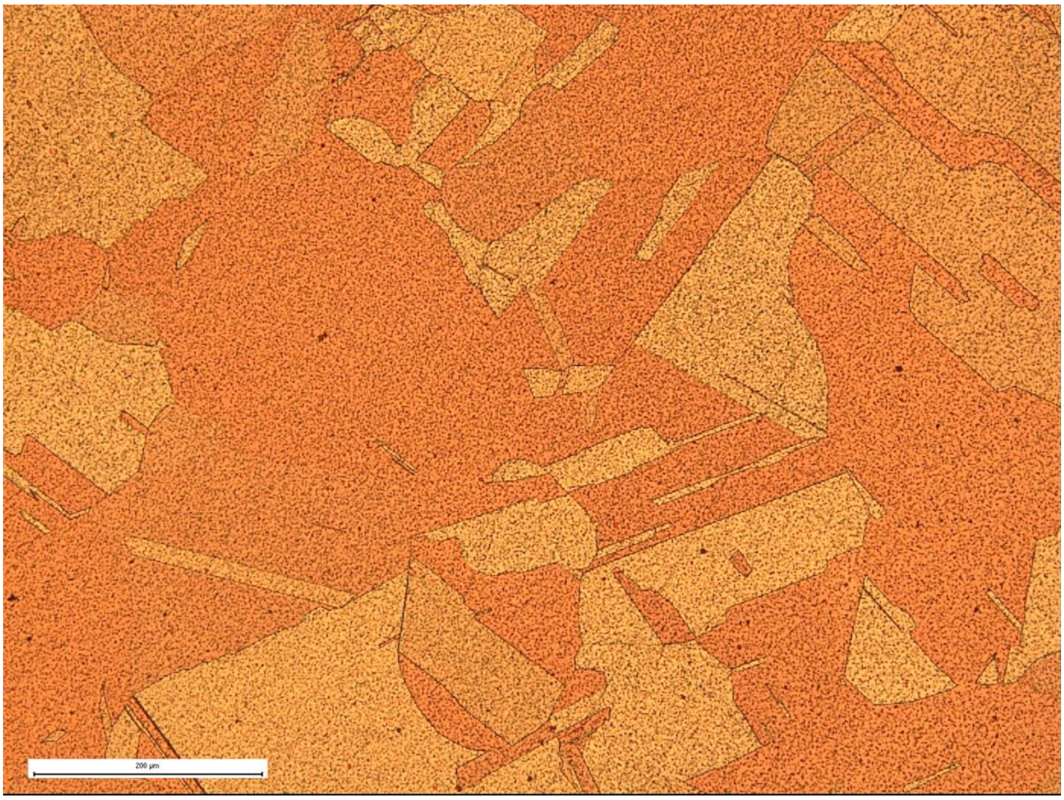

Figure 13. T58: Area A 100x.

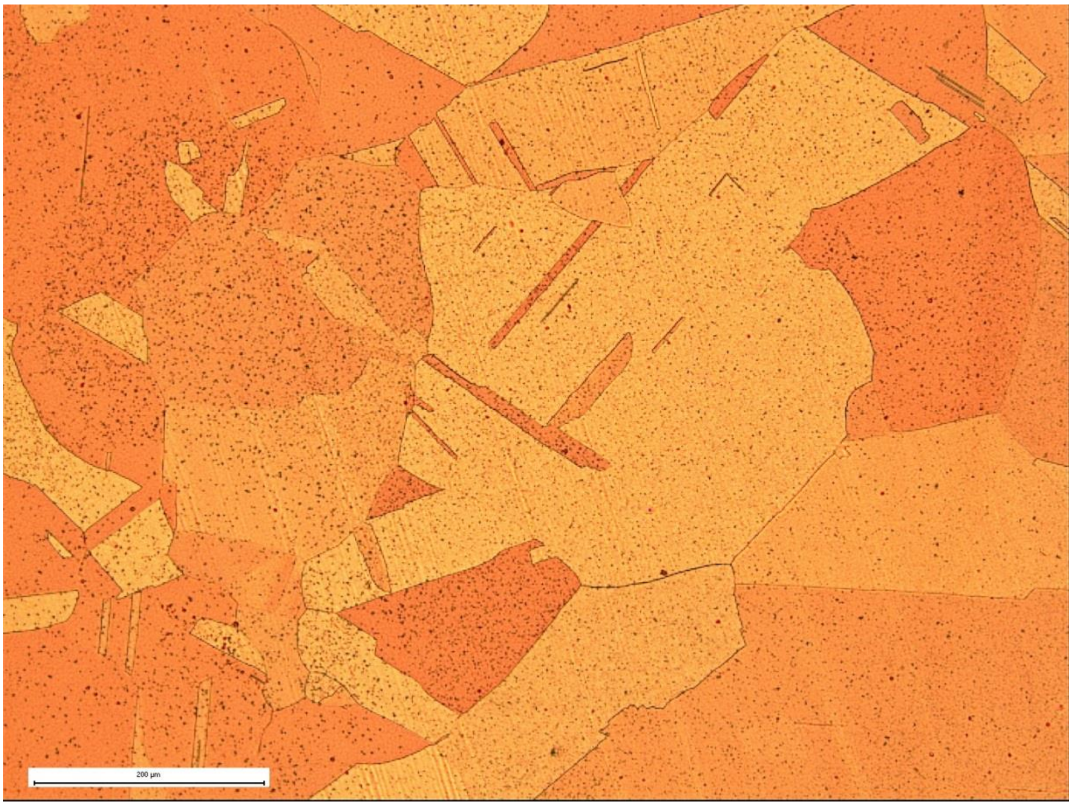

Figure 14. T58: Area B 100x.

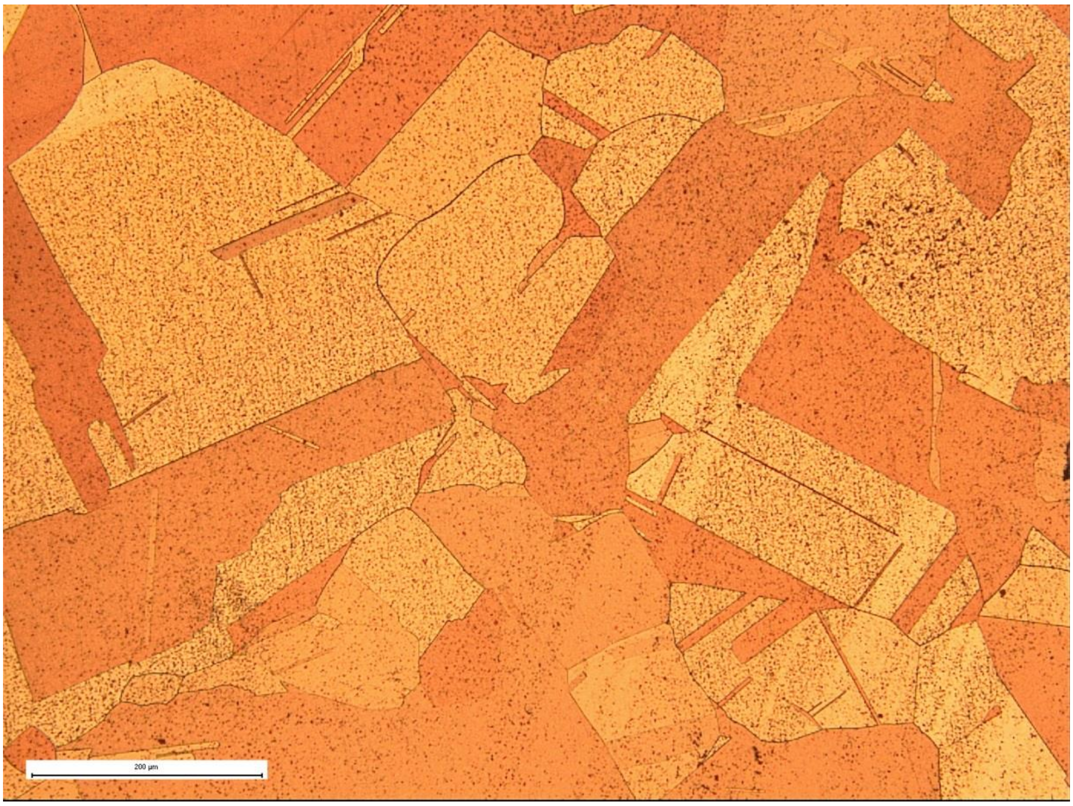

Figure 15. T58: Area C 100x.

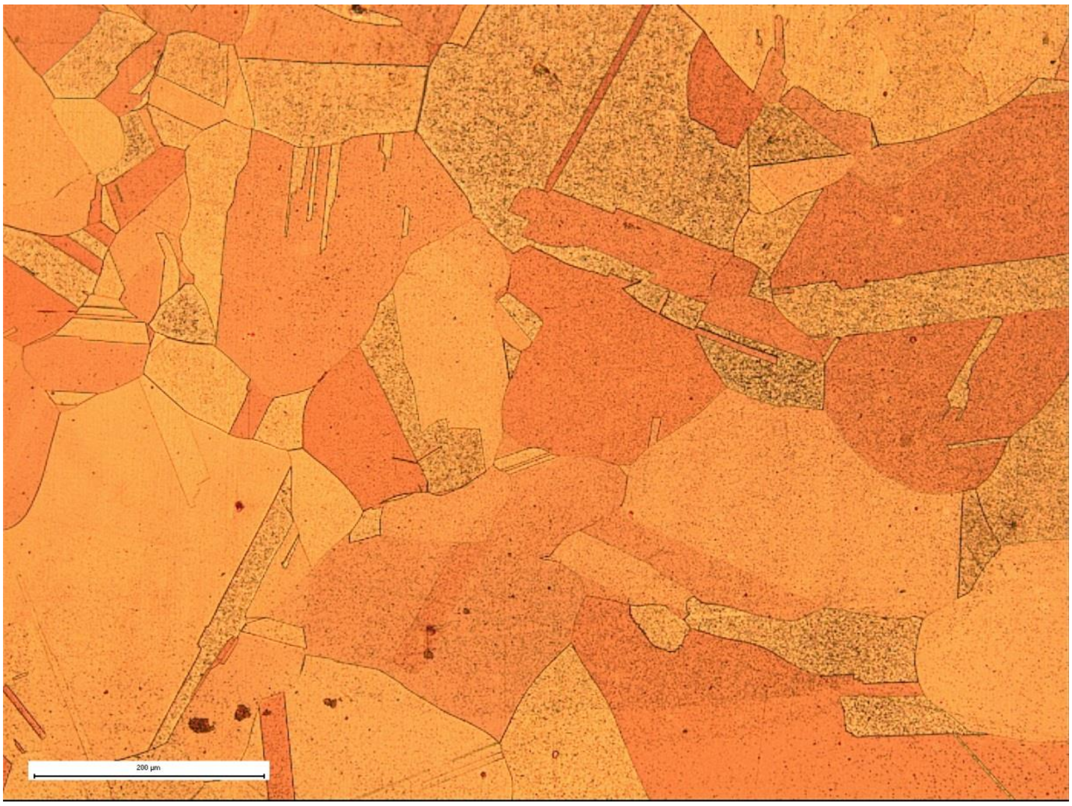

Figure 16. T64: Area A 100x.

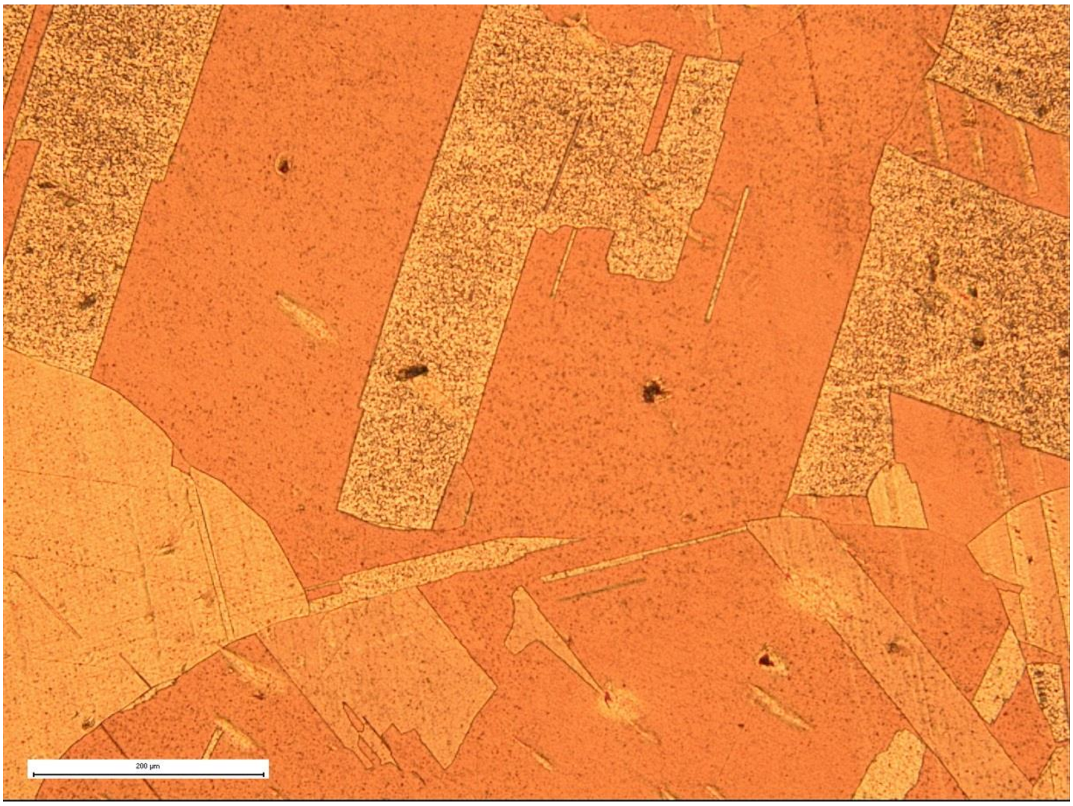

Figure 17. T64: Area B 100x.

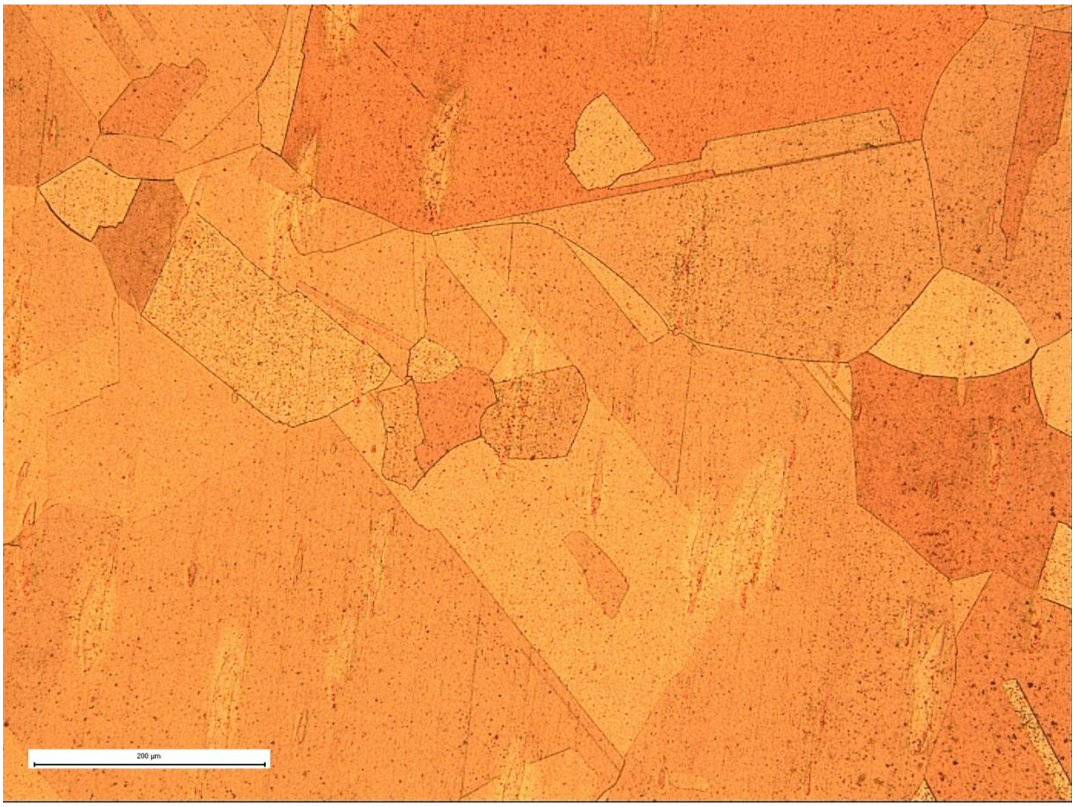

Figure 18. T64: Area C 100x.

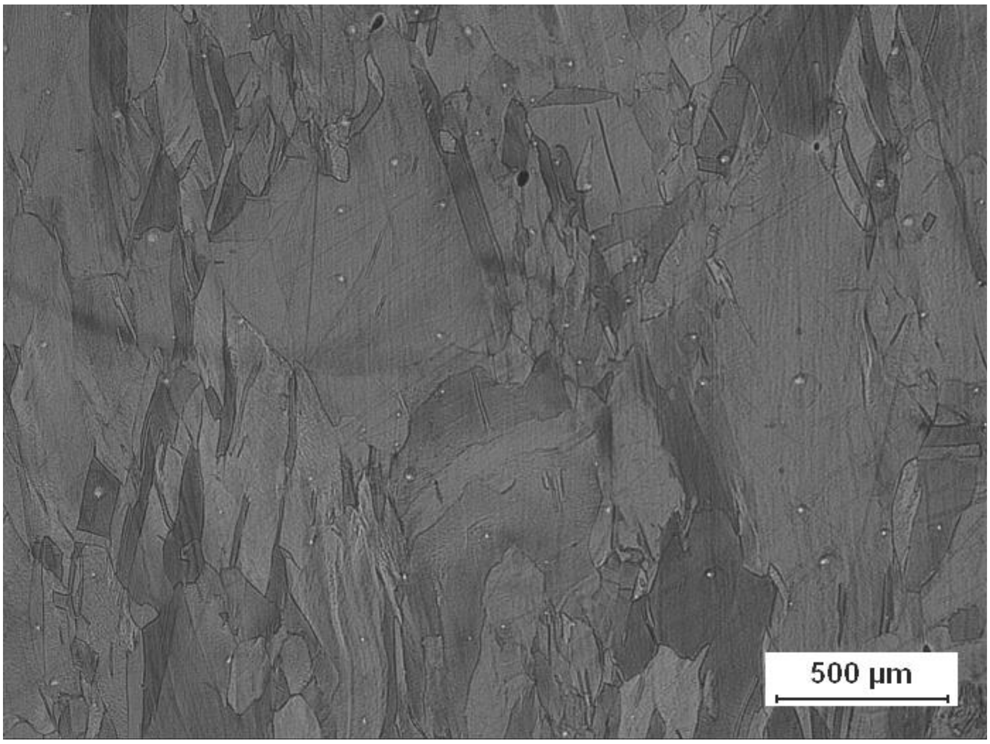

Figure 19. Micrograph of the structure in the gauge length of a specimen from the tube T53. The grains in the image is distorted due to the creep strain the material has experienced.(LOM 100x).

3.3 Hardness and chemical composition

The chemical composition for oxygen (O), phosphorous (P) and sulphur (S) is shown in Table 4, [2]. No significant difference, apart from in phosphorous, is observed. Table 5 shows the hardness for the three different tubes. The difference between the extruded tubes T52 and T58 and the pierced-and-drawn tube T64 is evident.

Table 4 The O, S and P levels in T53, T58 and T64[2].

	T53	**T58**	**T64**	**Method**
	(ppm)	(ppm)	(ppm)	
O	2.3	3.2	2.3	3
P	72	54	49	1
S	< 5	< 5	< 5	2

1 Spectroscope, ARL 3460
2 Melt extraction, Leco CS 225
3 Melt extraction, Leco TC 436 DR

Table 5 The hardness values measured in T53, T58 and T64 [2].

Name	**Hardness**
	HV5 (std dev)
T53	40 (0.7)
T58	40 (0.6)
T64	48 (1.2)

4. Discussion

The copper studied in this work came from areas in the copper tubes that exhibited higher sound attenuation than normal during ultrasound testing. The difference in attenuation could result from grain size variations or from segregations of alloying elements. The results from the parallel investigation on oxygen, phosphorous and sulphur variations in the same materials, [2] show that the difference in alloying levels is low and within the set thresholds. The same investigation also shows that the hardness variations between the materials studied is low. The only other reason that could be behind the higher sound attenuation is grain size.

The studies of the grain size in creep specimens show that the grain size does not vary much between the three tested tubes. The distribution of small and large grains is different though with the extruded tubes having a mixture of small and large grains, and the pierced-and-drawn tube only large grains. The large grains are of the same size in all materials.

Notwithstanding the grain size variations, the creep response is similar for all the materials. Especially if the fact that one set of specimens is taken from a pierced-and-drawn tube and the other two from extruded tubes. This yields a difference in the amount of cold work introduced in the material during manufacturing of the tubes, and this is shown in the creep results. That there is a difference in the amount of cold deformation is evident from the previously mentioned hardness studies. The difference is small but considering that every precaution was taken in this work to limit the amount of cold deformation introduced during manufacturing of the specimens, i.e. the spark eroded gauge lengths, it is likely that the difference will show up in creep testing. It is therefore, in our opinion, likely that the grain size variation is not affecting the creep results, and that the results shown are down to the amount of cold work introduced during tube manufacturing.

In addition to this all tested specimens exhibit large ductility. The loading strain in most specimens is around 20% and the creep strain around 15-25%, giving a total strain of mostly around 45-50%. Area reduction is in the region of 60-80%, which for this type of material are normal values. At first glance the area reduction might seem slightly lower than previous, but this is down for two reasons. The first is that the cross-section is not round as before but square, which means that the measurements cannot be conducted in the same way. Also the tests are for technical reasons not allowed to fully fracture, but are stopped about 30 seconds before fracture. Thus the two parts of the specimens are still connected and this might mean that the reading of area reduction is lower. The necking does however still has the same shape as in previous creep tests and the ductility should be the same if the part had been allowed to separate.

The Norton plots show that the creep exponents fall between 20 and 67, which is well into the power law break-down area as defined in the literature, e.g.[4]. This is also consistent with previous testing of similar materials.

5. Conclusions

Testing has been performed on copper, which, in non-destructive ultrasound testing, has shown higher than normal sound attenuation. Some conclusions from the work:

- No significant difference in creep response between higher sound attenuation copper and standard copper can be observed.
- The grain size in the extruded tubes is similar with a mixture of large and small grains. The pierced-and-drawn tube has a grain structure consisting of only large grains.
- The slight difference in creep response in copper specimens taken from extruded tubes and pierced-and-drawn tubes can probably be attributed to the difference in cold deformation introduced by the tube manufacturing method.
- All specimens show normally high values of ductility when compared to previous investigations.

6. Acknowledgment

The Swedish Nuclear Fuel and Waste Management Co (SKB) is gratefully acknowledged for funding this work and supplying the test material. The authors would also like thank Mats Larsson for help with the experiments.

References

1 SKB TR-10-14, Swedish Nuclear Fuel and Waste Management Co, Box 250, SE-101 24 Stockholm (2010)

2 Andersson-Östling H.C.M, ” Measurements of hardness and P, S and O levels in copper”, KIMAB-2013-123, Swerea KIMAB AB, Box 7047, SE-164 07 KISTA, Sweden (2013), SKB doc 1409401 rev 1.0

3 Andersson-Östling H.C.M., Sandström R., “Effect of loading rate on creep of phosphorous doped copper”, SKB TR-11-09, Swedish Nuclear Fuel and Waste Management Co, Box 250, SE-101 24 Stockholm (2011)

4 Evans R.W., Wilshire B., ”Creep of metals and alloys”, Book 304, The Institute of Metals, 1 Carlton House Terrace, London SW1Y 5DB, ISBN 0 904357 59 7 (1985)

5 Z. Jefferies, A.H. Kline, E.B. Zimmer, The determination of grain size in metals, Trans. AIME, 57 (1916), pp. 594–607

Swerea KIMAB is a leading corrosion and metals research institute. Swerea KIMAB develops and improves materials and processes for material production and studies the behaviour of new materials in process engineering and mechanical structures.

Research and development work takes place in close co-operation with Swedish and international companies in the steel, metals, electronics, engineering, paper, vehicle, manufacturing, plastics and power industries. For our customers, the results of our work should be a good investment for future revenue.

swerea | KIMAB

Swerea KIMAB AB
Box 7047 SE-164 07 Kista, Sweden
Tel +46 (0)8 440 48 00, Fax +46 (0)8 440 45 35,
kimab@swerea.se, www.swereakimab.se