REPORT

ASSESSMENT OF MECHANICAL PROPERTIES OF RRR300 NIOBIUM AFTER COLD WORK AND HEAT TREATMENT

Abstract

The following document details the experimental campaign and analyses to be performed in collaboration with Miskolc University (HU) in order to improve the current understanding of the mechanical properties for RRR300 niobium after processes typicaly performed durign the production of SRF cavities.

TRACEABILITY

Prepared by: A. Gallifa Terricabras [EN-MME-MM] and A. Amorim Carvalho [EN-MME-EDM]	***Date:*** 2018-12-03
Verified by: I. Aviles Santillana [EN-MME], L. Dassa [EN-MME]; E. Cano Pleite [EN-MME]; M. Garlasche [EN-MME]; N. Valverde Alonso [BE-RF]	***Date:*** 2018-12-12
Approved by:	***Date:*** 20YY-MM-DD
Distribution: N. Surname (DEP/GRP) *(in alphabetical order)*	

Rev. No.	***Date***	***Description of Changes*** *(major changes only, minor changes in EDMS)*
0.5	2018-12-12	First version

1 INTRODUCTION AND CONTEXT

The present study has been launched in the framework of High Luminosity Large Hadron Collider (HL-LHC) project and within the Crab Cavity work package in order to assess the mechanical behaviour of ultra-high purity niobium after cold forming operation and additionally, after cold forming and subsequent heat treatment.

The aim of the study is to better understand the mechanical properties that the material achieves after manufacturing operations (including heat treatment) and during its final application at ~2 K.

Due to the difficulties in extracting tensile specimens compatible with the standards in force from a deep drawn part, a tensile test campaign has been foreseen using niobium specimens submitted to different degrees of controlled deformation (via cold rolling) and heat treatment (650 °C for 24 hours under vaccum). The initial thickness of the specimens is 4 mm.

For the cold rolling operation, CERN has contacted the Institute of Physical Metallurgy, Metalforming and Nanotechnology from Miskolc University (HU) which has extensive experience in the domains of cold rolling and metal forming, in order to launch a collaboration for the present study.

In parallel, hardness tests and metallographic/texture analyses will be performed on niobium specimens stemming from components which have been submitted to the real deep drawing operation. These results will be used to comparethe hardness and texture of the specimens which have been cold rolled and heat treated at Miskolc University.

2 PRELIMINARY TESTS[1]

Ideally, CERN requires that Miskolc University checks the proper rolling parameters (force, speed, etc.) to achieve (for a given thickness reduction):

- Equal deformation in both directions of the plane
- Equal deformation along the trough thickness (to the greatest extent possible)

For that, CERN requires that Miskolc University performs the following checks on three preliminary samples:

- Assessment of the microstructure in the through thickness in the rolling direction 1 and rolling direction 2 by checking, after forming, the stretching of the grains shape in the rolling directions and the compression of the grains shape along the thickness.
- Hardness profile along the thickness for the 2 rolling directions. Correlate the results with hardness measurements on the top surface of the sheets.

Other preliminary tests like friction of rolling and selection lubricant type are expected to be done by University of Miskolc.

[1] To be agreed upon the parts.

2.1 Additional information for the preliminary tests

For the preliminary tests previously mentioned, CERN has sent 3 samples of dimensions 100x50x4mm (see Figure 1):

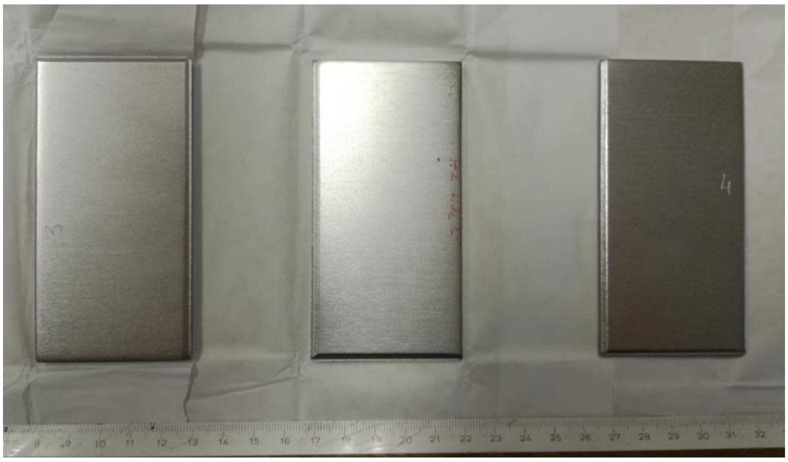

Figure 1. Nb sheet samples for preliminary tests.

From CERN experience, please note that niobium is a material prone to galling (or adhesive wear) during forming operations.

Note also that the current set up for forming operations with Nb at CERN includes a urethane film (for further information see https://www.acrotechinc.com/acro-hyde-die-film/) of about 0.4 mm thickness between the tools and the Nb parts. A lubricant called 'suif industriel' from the brand 'REACTOLAB' is also added to all surfaces.

3 TESTS CAMPAIGN

In order to study the influence of different degrees of cold work in the mechanical properties of niobium, several sheets will be cold rolled by Miskolc University until achieving the thickness reductions presented in Table 1.

In addition, to study the influence of the heat treatment required for the production of the Crab Cavities, tensile tests will be conducted to specimens (after the corresponding thickness reduction by cold rolling) before and after the heat treatment (650 °C for 24 hours under a vacuum level of ~$5x10^{-6}$ to ~$1x10^{-7}$ mbar[2]).

Tensile tests at room temperature (RT) and at cryogenic temperature (~4 K) are foreseen in order to assess the mechanical properties of the SRF cavities at ambient temperature and during operation (~2 K). The 2 K difference between the testing temperature and the operation temperature does not have an impact in the mechanical properties and associated fenomena occurring during the tests.

[2] The vacuum level during heat treatment is less than $5x10^{-6}$ mbar when starting the heat treatment and in the range of 10^{-7} mbar at the end.

Table 1. Test matrix foreseen for the study. The number of specimens indicated in each cell corresponds to tensile tests.

	before HT		after HT	
Thickness reduction	RT	4 K	RT	4 K
0 %	3 specimens	2 specimens	3 specimens	2 specimens
X %	3 specimens	2 specimens	3 specimens	2 specimens
Y %	3 specimens	2 specimens	3 specimens	2 specimens
Z%	3 specimens	2 specimens	3 specimens	2 specimens

A full set of tensile tests will be performed, at first, on two thickness reductions set of samples (X % and Z %) in order to study the influence of the cold work and subsequent heat treatment on the mechanical properties of niobium. If this influence is confirmed, Y % will also be fully studied.

However, enough material to perform all the thickness reductions specified in Table 1 will be cold rolled and heat treated, but the specimens will not be cut nor tested until the influence of deformation + heat treatment is confirmed.

As complementary information, metallographic studies and hardness measurements will be performed to samples from each of the cells in Table 1. These last results will be compared to the hardness and metallographic analysis of samples extracted from the leftover of a part after real deep drawing operation.

In summary, **the tests foreseen for Nb sheets after cold rolling at several thickness reductions and heat treatment**, according to Table 1 are:

- Tensile tests at RT (3 samples, oriented at 0°) (to be discussed if needed at 90° and 45°)
- Tensile tests at 4 K (2 samples +1 backup, at 0°)
- Metallography to check grain flow distribution and grain size measurement (1 or 2 directions)
- Hardness profile along the thickness (1 or 2 directions)
- Dislocation density analysis or texture analysis (to be discussed) (1 or 2 directions or along thickness).

The tests, to be performed at CERN, on **samples extracted from parts which have seen the real deep drawing** operation are:

- Metallography to check grain flow distribution and grain size measurement (1 or 2 directions)
- Hardness profile along the thickness (1 or 2 directions)
- Dislocation density analysis or texture analysis (to be discussed) (1 or 2 directions or along thickness).

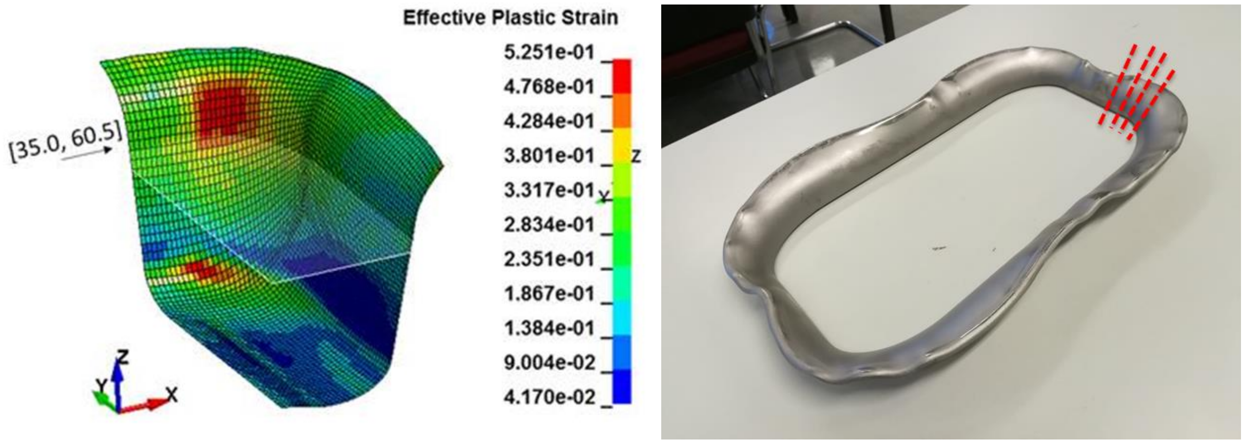

Figure 2. (Left) Simulation of a quarter of one pole of RFD cavity, with colour scale according to level of Effective Plastic strain reached after forming. (Right) Leftover from RFD pole after forming (made in niobium RRR300).

4 MATERIALS AND PREPARATION OF THE SPECIMENS

The material used for the study includes the following:

- Niobium RRR300 sheet 4 mm thickness. (maximum foreseen dimensions needed for the whole study: 600x600x4 mm, see Figure 3). The sheet shown in Figure 3, will be divided in 9 sub-sheets (8 for tests + 1 backup). 4 sub-sheets will be tested with no heat treatment and 4 sub-sheets after heat treatment. 6 sub-sheets need to be cold rolled at the specified X%, Y% and Z% thickness reduction.

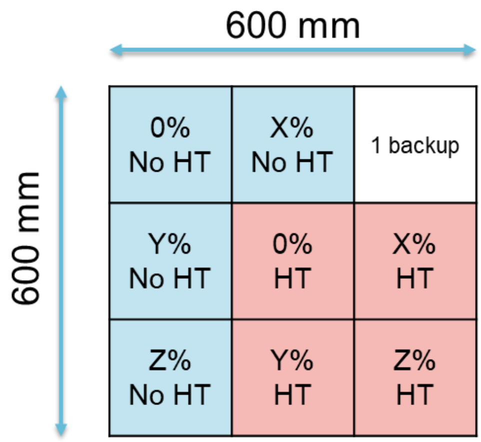

Figure 3. Scheme of maximum foreseen amount of Nb sheet material needed.

- Leftover parts after deep drawing (leftover from RFD pole forming, see Figure 4). Both horizontal and vertical specimens will be extracted from the pole leftover, which will be extracted in the zones where the maximum deformations are expected (see Figure 2).
- [Please refer to chapter 2.1 for the size and quantity of the material for the preliminary tests]

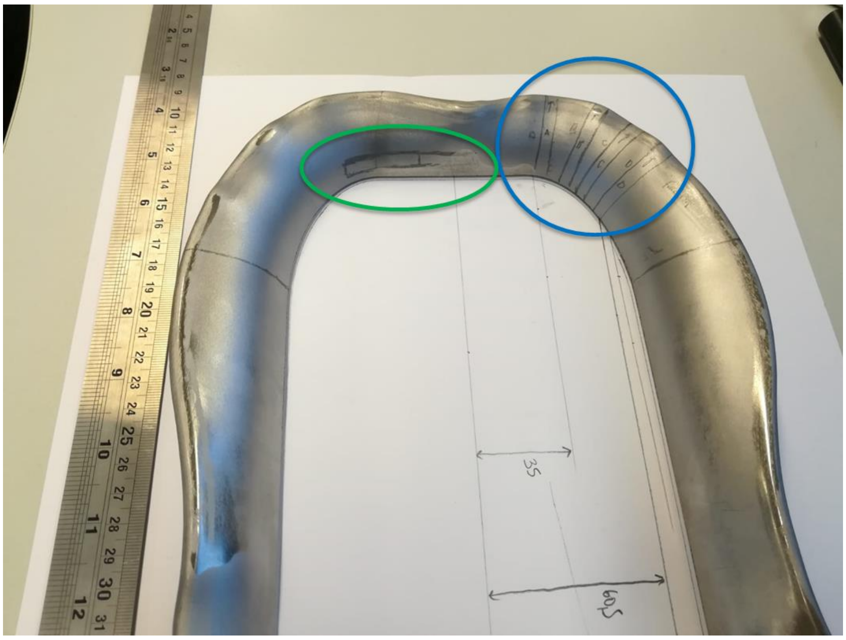

Figure 4. Specimens to be extracted from the RFD pole leftover after forming: marked in blue the 'vertical specimens', marked in green the 'horizontal specimens'.

5 FLOW CHART OF TASKS FORESEEN AND PLANNING

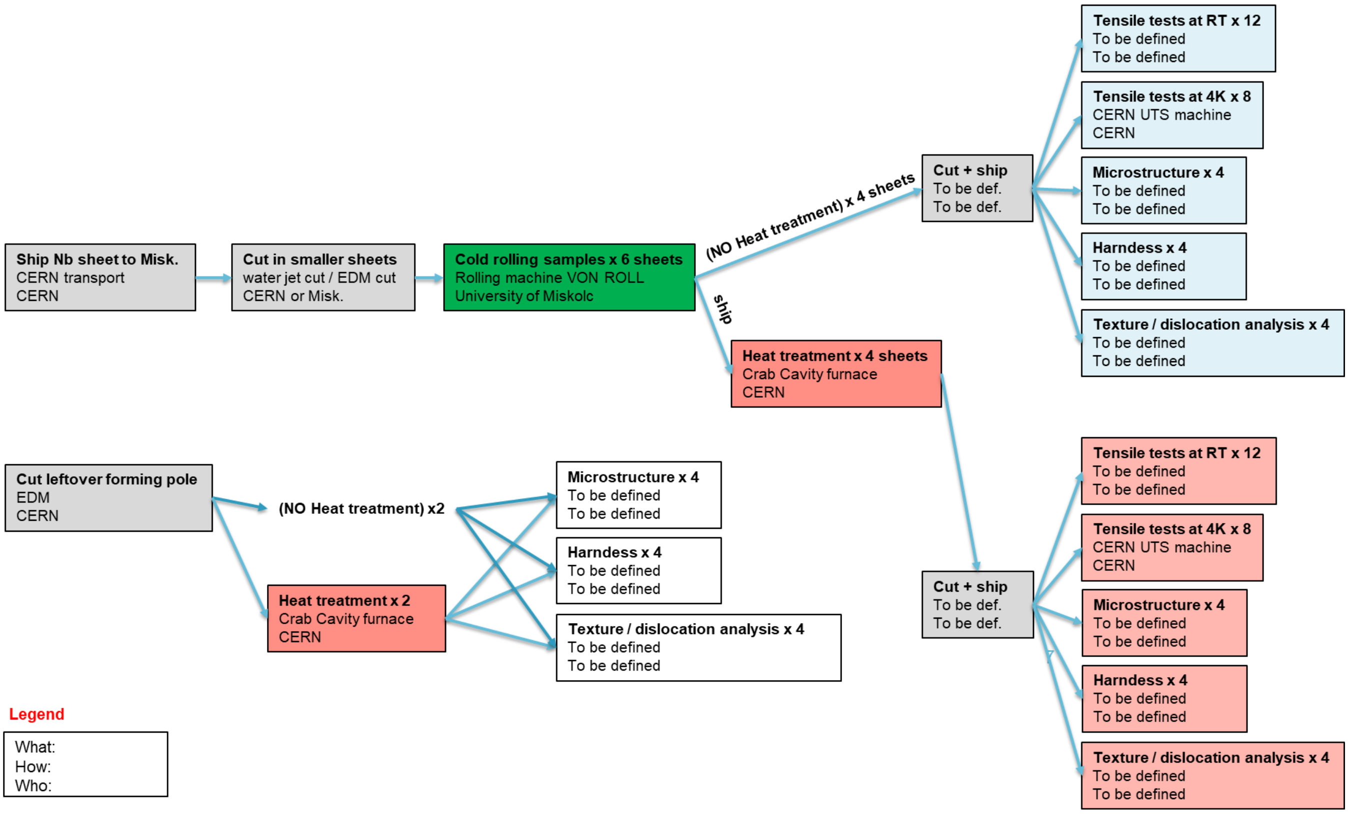

Figure 5. Flow chart of the tasks to be performed during the collaboration.

The list of tasks for the part of the study concerning the rolled sheets is the following:

Activity description	Responsible	Activity ID	Preced.	Estimated duration time	Expected timming
Material reception + Ultrasonic testing	CERN	0		-	T0
Ship Nb sheet to Miskolc university	CERN	A		2 weeks	T0 + 2weeks
Cut sheet in smaller sub-sheets	To be defin.	B		2 weeks	T0 + 4weeks
Cold rolling samples x 6 sub-sheets	Miskolc uni	C		5 weeks	T0 + 9 weeks
Cut samples (after rolling and without HT) into specimens	To be defin.	D		2 weeks	T0 + 11 weeks
Ship rolled specimens to CERN to do HT and/or tests	Miskolc uni	E		2 weeks	T0 + 13 weeks
Perform Heat Treatment (HT)	CERN	F		3 weeks	T0 + 16 weeks
Cut samples (after rolling and HT) into specimens	CERN	G		2 weeks	T0 + 18 weeks
Ship samples (after HT) to Miskolc university to perform tests	CERN	H		2 weeks	T0 + 20 weeks
Tensile tests RT (with no HT) x 12 specimens	To be defin.	I		3 weeks	
Tensile tests 4K (with no HT) x 8 specimens	CERN	J		4 weeks	
Microstructure (with no HT) x 4+ specimens	To be defin.	K		2 weeks	
Hardness measurements (with no HT) x 4specimens	To be defin.	L		2 weeks	
Texture/dislocation analysis (with no HT) x 4specimens	To be defin.	M		8 weeks	
Tensile tests RT (after HT) x 12 specimens	To be defin.	N		3 weeks	
Tensile tests 4K (after HT) x 8 specimens	CERN	O		4 weeks	
Microstructure (after HT) x 4 specimens	To be defin.	P		2 weeks	
Hardness measurements (after HT) x 4 specimens	To be defin.	Q		2 weeks	
Texture/dislocation analysis (after HT) x 4 specimens	To be defin.	R		8 weeks	

6 ANNEX A: TENSILE SPECIMEN FOR ROOM TEMPERATURE TESTS (TO BE COMPLETED)

7 ANNEX B: TENSILE SPECIMEN FOR CRYOGENIC TEMPERATURE TEST (TO BE COMPLETED)