HYDROFORMING OF NIOBIUM RF CAVITIES : MECHANICAL PROPERTIES OF Nb IN RELATION TO ITS FORMING ABILITY

C.Z.ANTOINE, D. COCHET
CEA, DSM/DAPNIA/Service d'Etude des Accélérateurs
CE-SACLAY, F-91 191 Gif-sur-Yvette Cedex

SUMMARY

Mechanical properties of niobium samples have been measured after various thermomechanical treatments : rolling, annealings at different time and temperature. Comparison with data from literature is made, and the accuracy of the different measurable parameters is discussed in view of RF cavity fabrication.

INTRODUCTION

Hydroforming is one of the possible ways to industrially produce seamless cavities, which is an advantage in terms of costs and reliability. This idea still need a feasibility demonstration, and several laboratories and/or companies are on this "track".

To achieve such high deformations of Nb tubes as required for forming a cavity (about 250 %), one really need to know which parameters are important in the forming process, and how they can be optimised.

In term of mechanical properties of the niobium, we can sum up the situation with two kinds of criteria :

- **1st order criteria :** we want to have the most important deformation ("elongation" in the case of traction curves) as possible before breaking the material. Actually, what we need is the deformation before necking, especially if we need to handle this deformation into several steps, because after necking the thinning of the material becomes unstable, and breaking can occur at any localised defect. We shall use the term " uniform " for this type of deformation at the difference with total (maximum) elongation that is measured at breaking
- **2nd order criteria :** we need to preserve a good surface state and purity.

Several parameters can influence those criteria, some linked to the niobium itself, and some linked to its metallurgical state.

Parameters linked to the metal :

- Purity : like every metals, mechanical properties of niobium depend on materail purity : the more pure the softer is the material is. But we shall show hereafter that within the range of purity that we handle, this effects is second order compared to the actual metallurgical state of the metal.
- Speed sensitivity : this point is very particular to BCC (Body Centered Cubic) metals ; several studies, including ours show that the reachable homogenous elongation depends on the strain rate and present a maximum value.

Parameters linked to the metallurgical state :

It is very difficult to fully characterise the mechanical state of a piece of metal. There exist overabundant amounts of mechanical tests (traction, creeping, torsion, deepdrawing with special forms of blanks, etc...). Each one enlightens a specific behaviour of the material in given conditions. They allow to draw what is called a " Forming Limit Diagram " in the plane of bidimensionnal strains, which for instance is currently used to predict behaviour of blanks even for complex deep drawing processes. (For examples of FLD, see ref [1] for Niobium, and [2] for a more complete overview). But as this FLD is difficult to get (need of many testing facilities and material sample), the most common test used to shortly describe the behaviour of a material through a simple deformation process are :

- Hardness : this gives the general trends of the mechanical behaviour of the material, in particular, it a very practical and quick way to obtain information on the changes that occur with a thermo-mechanical treatment (cold working , recovering, recristallisation....), although it is not very precise. (Hardness is also sensitive to purity, but in a lesser extent).
- Traction curves give us information about the behaviour of the material in a very specific way of deformation. Parameters determined with such a test are very commonly used to characterise deformation in general because it is very drastic to the material, and figure out somehow the "worse" conditions that can apply to the material
- Metallography gives information about the structural state of the material, in particular about the homogeneity, and the size of the grains, which have very important influence on the final result (thickness homogeneity, roughness, surface state).

It is well known that metals that have a "good" forming behaviour, should exhibit the following feature when performing the precited tests (within the range of variation corresponding to the type of metal of course!) :

- A low hardness
- A uniform and rather small grain size to accommodate better local deformations
- And the traction curve must exhibit the following parameters :
  - A low tensile strength, which implies lower efforts of forming
  - An enhanced elongation, which is of course the main parameter, as it traduces the feasibility of the deformation into a few steps. One should note that the parameters that is of interest in the case of forming is the "uniform" elongation before necking and not the ultimate elongation.
  - An enhanced strain hardening coefficient **n**. The influence of this parameter is also very important because it traduces the resistance of the material toward local instability (i. e. necking) : indeed, when some local deformation occurs, this area of the material is "hardened", and then becomes more (as more as n is high) resistant to deformation. Then the other softer parts of the material will start to deform first under strain. The more n is high, the more deformation can occur uniformly inside the whole material, and the later localised necking appears.

Note : the experimental measurement depends on eventual textures induced by deformation and can vary with the samples orientation.

All these parameters vary a lot with the thermomechanical story of the material, especially- among the BCC metals like niobium. Only well recristallized material can exhibit such feature. In the following we show some results that were measured on niobium dedicated to the production of welded tubes in order to hydroform them.(supplier = TELEDYNE, RRR = 370) The goal is to determine in what condition we can recover "good" forming parameters after the deformations the sheets, and/or the tubes will encounter. Additional data from the literature are added for comparison.

EXPERIMENTALS

sensivity to purity :

In reference [2] ,a very complete comparison is made between four different purities of niobium : " commercial ", " intermediate purity ", " high purity ", " ultrapure ", all in the same very well recristallised state, with the same grain sizes. Comparison of the announced composition indicate that commercial quality corresponds to a RRR less than 10 while ultrapure correspond to RRR better than 300. One can see on table 1 that homogenous elongation measured on these samples varies between 23 and 30 % with purity, and when the niobium becomes quite pure, this variation is not very sensitive. The same measurement were made on a very pure sheet of niobium (RRR ~ 370) at different stages : as received, 46 % deformed (rolled), annealed without full recrystallisation and then fully recristallised. The most meaningful parameter : the uniform elongation εu is quoted in table 1 ; between the two last steps the difference is already huge : 2.6 to 30 % !!!

Table 1 : influence of purity compared with influence of metallurgical state on εu (elongation at necking)

Nb Type of sample reference [2]	εu %	Nb RRR 370 Type of sample [this work]	εu %
" commercial "	22 %	as received	26 %
" intermediate purity "	27 %	46 % rolled	0.5 %
" high purity "	30 %	46 %+600°C, 2 h a)	2.6 %
" ultrapure "	31 %	46 %+800°C, 2 h b)	30.6 %

a) beginning of recristallisation. b) recristallisation completed

This simple comparison indicates that purity is not the main factor to achieve good forming properties, and for financial reasons it is worthwhile to foresee a postpurification of the material, and to start with less expensive low RRR material.

sensitivity to strain rate :

Figure 1 shows the dependence of maximum elongation of three different niobium batches (although quite close in composition) versus strain rate as found in references [2]and [3] ,and as measured in our laboratory. One can notice that all the experimental figures are very close, but this sensitivity is quite important : one can gain about 10% (from 20% to 30%) in elongation by judicious choice of the strain rate. We assigned the optimum value of 5.10^{-3} S^{-1} for the strain rate of every following deformation experiments.

M : Niobium supplier : Pechiney, data from P. Mazot, PhD Thesis, Poitiers, 1970 [ref 4]
H : Niobium supplier : Kawecki, data from C. Margoli, CERN Technical Note, ISR-GE/CM/fa, GE/81-15, 1981 [ref 5]
S : this work, Niobium supplier : Teledyne Wah Chang.

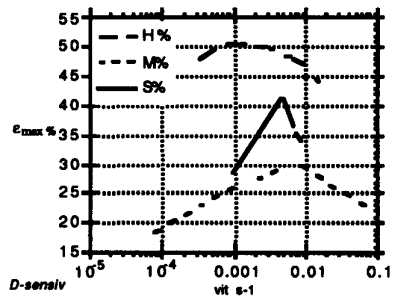

Figure 1 : dependence of maximum elongation of three different niobium batches versus strain rate

sensitivity to annealing conditions :

Annealing of the traction samples were conducted in an experimental oven, between 600° C and 900° C, during 2 or 4 hours. This oven is composed by a quartz tube linked to an Alcatel CFF 450 Turbo pumping group : a turbomolecular secondary pump linked to a primary oil pump. The vacuum during annealing is better than 10^{-3} Pa (10^{-5} Torr). This vacuum is typically not sufficient to keep RRR grade to the niobium, but the easiness and swiftness of use of this small oven led us to use it for a feasibility demonstration.

Niobium RRR 370	sample	Hv	Ø (µm)	σ_M MPa	ε_u(%)	σ_E MPa	n	sample	Hv	Ø (µm)	σ_M MPa	ε_u(%)	σ_E MPa	n
Without Heat Treatment	as received	56	45	151.2	26.1	120	0.096	rolled at 46%	64	-	241.3	0.5	238	0.196
Heat Treatment (Nb rolled at 46%)	2H							4H						
600°C								A		60	190.1	4.7	159	0.080
	B	64	44	197.2	2.6	163	0.075	B			199.7	2.4	176	0.119
								C		90	181.5	5.9	158	0.079
								D	73		195.6	2.5	166	-
700°C	A	64	60	172.5	9.8	137	0.116	A	69	60	161.4	11.2	134	0.089
								B		60	172.2	8.7	139	0.091
	C	64	110	175	10.6	142	0.104	C			167.5	11.8	131	0.114
								D			170.5	5.5	148	0.085
750°C								A		60	145.8	26.9	70.1	0.235
	B	52	60	157.6	26.0	82	0.198							
								C		60	155.9	20.6	99.7	0.157
800°C	A		160	146.8	30.6	64.5	0.283							
								B	36	310	147.8	31.1	57.5	0.272
								D	36	125	155.8	25.1	67.8	0.267
900°C	C		310	145.4	30.8	62.8	0.287							

A, B, C, D refer to rolling and cutting directions(see fig 2). Mechanical properties are measured on an INSTRON 4507 traction facility from the Service de Recherche Metallurgie Appliquée from CEA/SACLAY, Hv is the Vickers hardness (1KG charge), Φ the grain mean diameter in µm, σM the ultimate strengh, σE the yield stress, εu the uniform elongation and n the strain hardening coefficient.

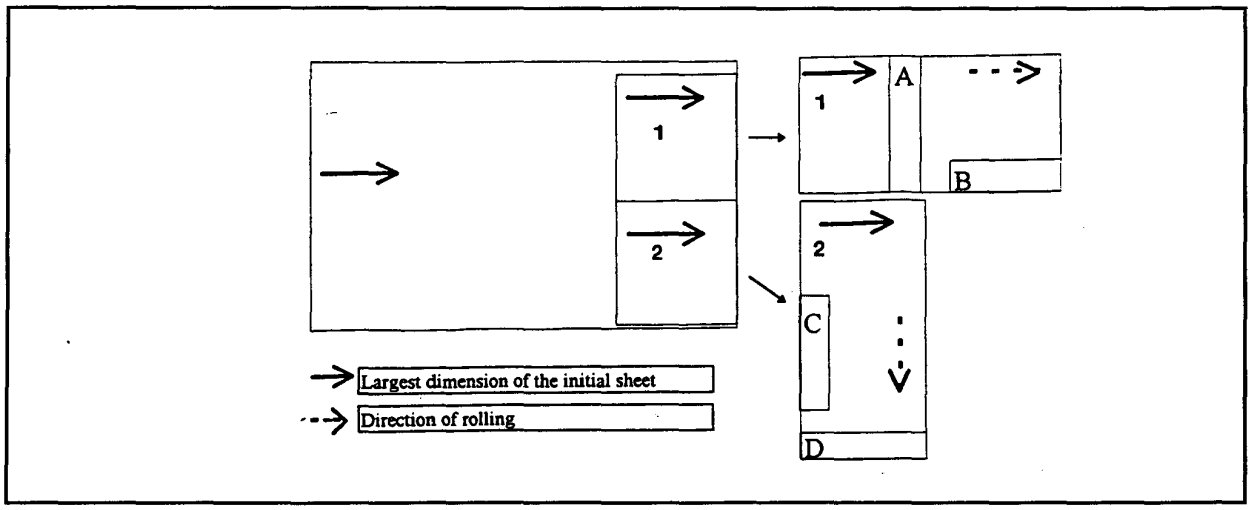

fig 2 : Description of the different orientations for rolling and cutting of the samples

Table 3 summarises some data obtained on niobium samples described in the introduction. One can see that recristallisation is very sensitive to temperature variations, and to a much lesser extent to annealing time. Although we can not control the final texture in the case of the tubes we also checked the influence of the rolling direction (four different " orientations " : A, B, C, D). The differences are quite important.

The important grain growth observed in our case seems an indication that temperature should be controlled to better than + 50° C. Quenching with rare gases is also very often evoked in the literature to control better the decrease of the temperature after heat treatment. One can note that although literature points out that recovering should occur before recristallisation [7] for niobium, this one is not very noticeable, and one has to wait until total recristallisation to reach good mechanical properties.

Uniform elongation measured on (800°C, 2 H), (800°C, 4 H), and (900°C, 2 H), are very close, but grain size tend to increase with time and temperature. Moreover the annealing parameters recommended by the company " TELEDYNE " is also very close to : (800°C, 2 H). Hardness measurements also indicate a noticeable transition at this time and temperature treatment. We shall then apply this treatment to niobium after its deformation steps.

Discussion on the strain hardening coefficient n : this coefficient is very often used in the " deepdrawing community ". Moreover, it as been proven in the case of biaxial deformation, that εmax varies like $4/11(2n+1)$ (ref [6]), i. e. that an increase of n favours biaxial deformation as it delays instability (necking) apparition (for more detail about this calculation and its discussion, see ref [6]).It is then obvious we should try to get an n coefficient as high as possible. Table 3 represents data from ref [6], compared with data about niobium measured in this wok or found in literature. Note that in the case of an easy formable metal like copper, n ranges between 0.3 and 0.45. The value found for niobium show that niobium can be as easy to deform as copper, and that n is not much influenced by impurities like O (samples 2 to 5 in table 3). On the over hand, this n value seems to be very affected by the metallurgical state of the metal.

Metal	n (ref [6])	Niobium	n
softened steel	0.15 - 0.25	a)	0.075 - 0.287
austenitic steel 18-10	0.4 - 0.5	b)	0.45
aluminium	0.07 - 027	c)	0.45
copper	0.3 - 0.47	d)	0.45
zinc	0.1	e)	0.45
nickel	0.6	f)	0.10
		g)	0.31

a) This work,
b) ref. [4], pure Nb ; c) ref. [4], pure Nb+ 80 Wppm O ; d) ref. [4], pure Nb+ 230 Wppm O ;
e) ref. [4], pure Nb+ 330 Wppm O ; All sample in a well recristallised state
f) Heraeus batch used to form half-cells, high failure rate at forming
g) Heraeus batch used to form half-cells, same purity, high success rate at forming
(Note :
- f) et g) exhibits very different feature on the traction curve, and can be explained by lack of control of the annealing.)
- when not explicitly found in reference, n is experimentally calculated with a graphic method described in ref [3] where n is the slope of the straight part of the curve $\ln(\sigma) = \ln(\varepsilon)$.

First experience with several deformation steps

At this stage of the study, it is necessary to check if it is possible to apply the annealing parameters for several deformation steps. Rolled samples where annealed 2 hours at 800°C, and then 25% deformed by traction, and then annealed again in the same condition. First experiments showed that it was not possible to get as much as deformation after the second annealing, and even less after the third one, and that it was difficult to recover a good strain hardening coefficient value. A second trial with 2 hours annealings at 900°C instead of 800°C showed the same behaviour, although with slightly higher elongation and strain hardening coefficient.

Several effects can conjugate to explain this disappointing result:

1) 800°C is maybe not sufficient to get total recristallisation in all the direction of the material

2) After several annealings, the grain size becomes very important and the mechanical properties are affected.

3) Damaging phenomena can occur and are not yet well studied on niobium

4) Although purity is not a 1st order parameter, it is certainly degraded after several annealings and the observed mechanical properties degradation can also be influenced by this.

CONCLUSION

It is obvious now that purity does not much influence the mechanical properties of niobium, but that a very good control of the recristallisation parameter is needed. These parameters are very much influenced by the thermomechanical history of the material, and are even more difficult to control as the material becomes very pure. Moreover complementary results are needed on the apparition and the nature of damaging phenomena when deforming niobium.

It seems then judicious in order to demonstrate feasibility of hydroforming of Niobium tubes into RF cavities to start with a less pure and less expensive material and to make efforts mainly on the control of its thermomechanical transformation from the earliest stage of fabrication as possible ; i. e. to concentrate specially on the preparation of the tubes.

From the point of view of deformation, it seems that the niobium studied in this work has not favourable properties, but one has to keep in mind that the deformation applied in the case of Hydroforming (more or less bidimensionnal expansion) is very different from the traction test (for instance the influence of the metal thickness is very important in the case of biaxial expansion and not very sensitive for traction). By the choice of appropriate tooling it is also possible to optimise the deformation in term of trajectory on the forming limit diagram of niobium. This last point is beyond the scope of this paper but shall be the next step of our approach.

ACKNOWLEDGEMENTS :

We would like to thank Mrs Janis, and MMr Le Naour and Tournié from the SRMA at Saclay for letting us free access to their traction facilities and helpful discussions.

REFERENCES

We apologise that many of this references are in French ; most of them are basic or advanced courses in metallurgy, and the equivalent should be very easily found in English.

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