FABRICATION OF NIOBIUM CAVITIES

W. Bauer
Kernforschungszentrum Karlsruhe, Institut für Kernphysik
P.B. 3640, 7500 Karlsruhe, Federal Republic of Germany

I. Introduction

For the application of RF-superconductivity in accelerators, storage rings and RF-separators [1] Niobium is most widely used as superconducting material. A few cases, where lead or Nb_3Sn were tried, will not be considered here. With one exception [2], all cavities were made of pure Niobium - either from solid material or sheet. The specifications for the Niobium used - often referred to as "Stanford - specifications" are shown in Table I.

TABLE I: Ordering Specifications for Niobium

Niobium "Reactor grade", purity 99,8%
Tantalum content less than 1000 ppm
Electron beam melted
cold forged
Sheets cross-rolled
Fully recrystallized

The Tantalum content used to be 300 ppm, but since last year the suppliers report difficulties about achieving this purity. The now quoted Ta content of 1000 ppm may have consequences for the heat transfer [4] because it reduces the thermal conductivity. A typical chemical analysis is shown in Table II. [3] It should be pointed out to the niobium supplier, that scratches on the sheet surfaces should possibly be avoided; covering the sheet by a protective paper layer before delivery would be very helpful to maintain smooth surfaces. The following report collects the methods for fabrication of cavities out of this material.

II. Mechanical Properties of Niobium

We start with a collection of mechanical properties of Niobium, the knowledge of which is necessary for designing and fabricating cavities. It is clear, that they depend very much on the state of the material, e.g. on the purity and on previous heat treatments (where the temperature, the time and the vacuum are important). Experience shows, that the behaviour of the material may differ without any noticeable change in specifications or chemical analysis, from one charge of material to the other. Very little is known about mechanical properties of Niobium at low temperatures.

Table II: Typical Analysis of "Reactor grade Niobium" [3]

CHEMICAL ANALYSIS IN PPM

	Top	Bottom
Al	<20	<20
B	<1	<1
C	40R,40R	60R,70R
Ca	<20	<20
Cd	<5	<5
Co	<10	<10
Cr	<20	<20
Cu	<40	<40
Fe	<50	<50
Hf	<50	<50
Mg	<20	<20
Mn	<20	<20
Mo	<20	<20
Ni	<20	<20
Pb	<20	<20
Si	<50	<50
Sn	<10	<10
Ta	415	480
Ti	<40	<40
V	<20	<20
W	56	53
Zr	<100	<100
O	<50	100
N	14	27
H	<5	<5

INGOT HARDNESS, BHN	
Average	55
Low	44.9
High	85.7

500 kg load

METALLOGRAPHY TEST RESULTS

Micro no: AM-310

Material is 100% recrystallized.

ASTM Grain Size Ave. no. (LONG.) 3.5

PRODUCT HARDNESS

50HV10

Table III can be used as a guide-line for the design of Niobium cavities. The data are collected from many sources [3,5-10]. For comparison, the values for pure oxygen free copper are also given, which show that in many respects Niobium can be handled like soft copper. Fig. 1 shows the strong influence of the oxygen content on the hardness. An important feature of Niobium is the fact, that it getters oxygen, but also hydrogen, carbondioxide, hydrocarbons and others at temperatures above 200°C. The brittleness caused by dissolved gases can be removed by a heat treatment in vacuum. Hydrogen can be outgassed at ≥ 800°C, for removing oxygen temperatures above 1500°C are needed. In addition, Niobium is always covered with an oxide layer.

Table III: Mechanical properties of Nb and Cu

Nb: purity 99.8%, electron beam melted, cold forged, recrystallyzed

Cu: Oxygen free, annealed, 99.95% purity

		Nb	Cu
density	g/cm^3	8.57	8.96
modulus of elasticity	$N/mm^2 \cdot 10^5$	1.05	1.2
tensile strength	N/mm^2	207-274	196-245
bending strength	N/mm^2	138	-
yield point	N/mm^2	20*-196	117
elongation	%	25-25	30-50
vickers hardness	N/mm^2	800**	400-500
melting point	°C	2468-2497	1080
heat of fusion	J/g	298.5	188.4
specific heat (300 K)	J/g/°K	.267	.375
recrystall. temp.	°C	830-1230	500
thermal expansion $(L_{4.2}-L_{273})/L_{4.2}$		1.43×10^{-3}	3.3×10^{-3}

* annealed several hours at 1800°C and 10^{-8} torr; ** see Fig. 1

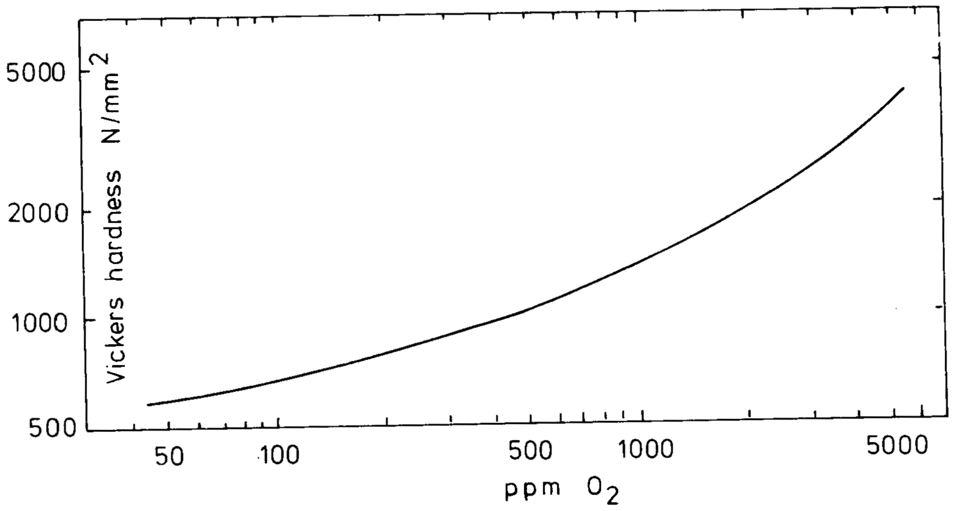


Fig. 1: Vickers hardness of Niobium versus Oxygen content[3]

Another property of pure Niobium is its ductility: It can be cold worked up to 99% without intermediate annealing. In practice, however, annealing in vacuum at ∿1000°C was found to be advantageous.

The conclusions from these properties for fabricating Niobium cavities can be summarized as follows:

- Shaping by machining, deep-drawing, spinning is possible, similar as for soft copper
- Welding needs a high temperature and much heat input because of the high heat of fusion and has to be done in completely oxygen-free environment, i.e. in vacuum or in a very clean inert gas atomosphere.

III. Machining

Machining tests have been carried through at all places, where Niobium cavities have been built. Machining Niobium needs much experience, especially in shaping the tool for free removal of the chips. Machining speed and cooling has to be adjusted to avoid heating up the material. Sometimes one charge of material is more difficult to machine than others. In some cases a previous heat treatment resulting in large grain sizes was reported advantageous, in others better results were obtained without heat treatment.

The tolerances achieved are of the order $\leq .01$ mm, i.e. as good as for other materials. In general deformations caused by heat treatment and welding have much more influence on the final tolerances than the initial machining.

Table IV. shows some parameters for machining which are recommended by Niobium suppliers and have been proven useful at many laboratories. They must be considered as a starting point, since every worker collects his own experience in shaping the tools and operating the lathe or mill.

As is shown in the table many lubricants or coolants have been tried, some of which underlying certain savety regulations like Trichlorethylene, which can only be used, if the lathe is equipped with an gas exhaust system. Although all other coolants give satifactory results it is still generally agreed that Tri should be used for very delicate surfaces and small parts.

The surface roughness achieved by careful machining can be as small as $\leq 5\mu$. In one case [11] a surface roughness of $0.2\ \mu$ has been reported.

Table IV: Parameters for Machining Niobium

Cutting tool:	High speed steel, sometimes also used: tungsten carbide (Widia) (only for fast light cuts, danger of breaking the tool).
Approach angle:	15^o - 20^o
Side rake:	20^o - 25^o
Side and end clearance:	5^o
Plan relief angle:	10^o - 20^o
Nose radius:	0.5 - 0.75 mm
Cutting speed:	20 - 25 m/min HSS 75 - 90 m/min Widia
Feed, roughing:	0.2 - 0.3 mm/rev
Feed, finishing:	0.01 - 0.1 mm/rev
Depth of cut, roughing:	0.75 - 3 mm
Depth of cut, finishing:	0.03 - 0.1 mm
Lubricants:	Trichloretylene, Tetrachlorcarbon, Chlorotene (safety regulations!) water, freon, air, oil
Achieved surface roughness:	$\leq 5\ \mu$

IV Sheetmetal Forming Techniques

Considering the fabrication of many cavities especially for low frequencies machining from solid is not possible for economical reasons. Several sheet metal forming techniques have been applied:

- Spinning: The sheet is fixed against a die on a lathe and rotated; then the material is pressed against the die by a handle. This method is used for single piece production, since the tools are relatively inexpensive.
- Deep-drawing: The sheet is formed in a hydraulic press between a die and a punch. This requires expensive tools and is the appropriate method for mass production.
- Hydroforming: The sheet is pressed into the die by hydraulic pressure of oil.
- Coining: The material fills after pressing the entire space between two tightly fitting dies.

1. Spinning

Experience in spinning Niobium exists at CERN[12], KEK[13], University of Wuppertal[14], and at the companies W.C. Heraeus[5], and Siemens[15]. The die is made of hard wood brass, bronce, steel, which is sometimes polished and hardened or aluminum anodized with a thick oxyde layer.

At CERN[12] the first spinning step is done using an aluminum die with the exact cavity dimensions. Then an annealing at 1000°C in a vacuum of 10^{-5} torr has been considered advantageous. After that the spinning is continued using a hard wood die with a slightly smaller diameter. Finally the piece is pressed into the aluminum die again and the spinning is completed.

Preferably the die should be on the inner cavity surface to achieve the best surface quality.

The handle can be brass or steel; sometimes it is equipped with a steel roll.

Periferal speeds of 150 - 1000 m/min are reported; the high speed is considered advantageous to reduce the need of intermediate annealing.

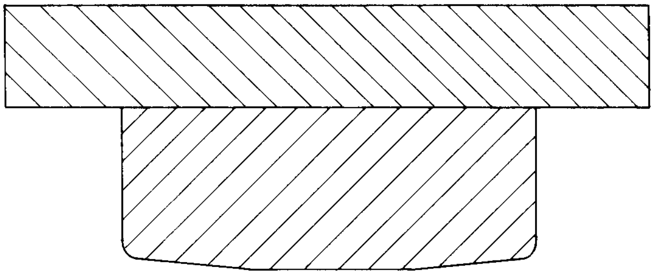
The tolerances achieved by this method are about 0.2 - 0.3 mm on a ∿ 600 mm diameter.

2. Deep drawing

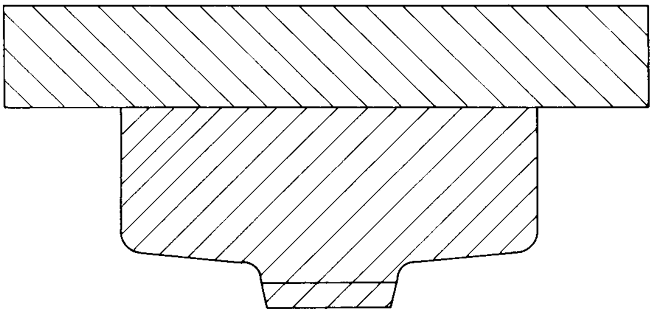
Deep drawing is used at Cornell[16], Genua[17], Interatom[18], Siemens[19] and others. Fig. 2a - 2c show examples for dies and punches used by Siemens for deep-drawing of separator-half-cells. If one aims at tight tolerances and good reproducibility one has to deal with the following difficulties:

- thickness tolerances of the material
- elastic properties of Niobium may change from on charge to the other
- removing the parts from the die is sometimes difficult and may result in dimensional changes. Experiments on suited die surfaces and lubricants might be necessary.
- in some cases machining of the inner surface after deep-drawing was considered necessary in order to achieve the tolerances and the surface finish required.

Table V shows a typical procedure, which was worked out at Siemens [19] for fabricating the CERN-Karlsruhe RF-particle separator. Later on this scheme was abandoned in favour of machining the whole cavity from solid. More recently Interatom [18] has built S-Band cavities for the University of Wuppertal[14] using a deep-draw technique without intermediate heat treatment or final machining.

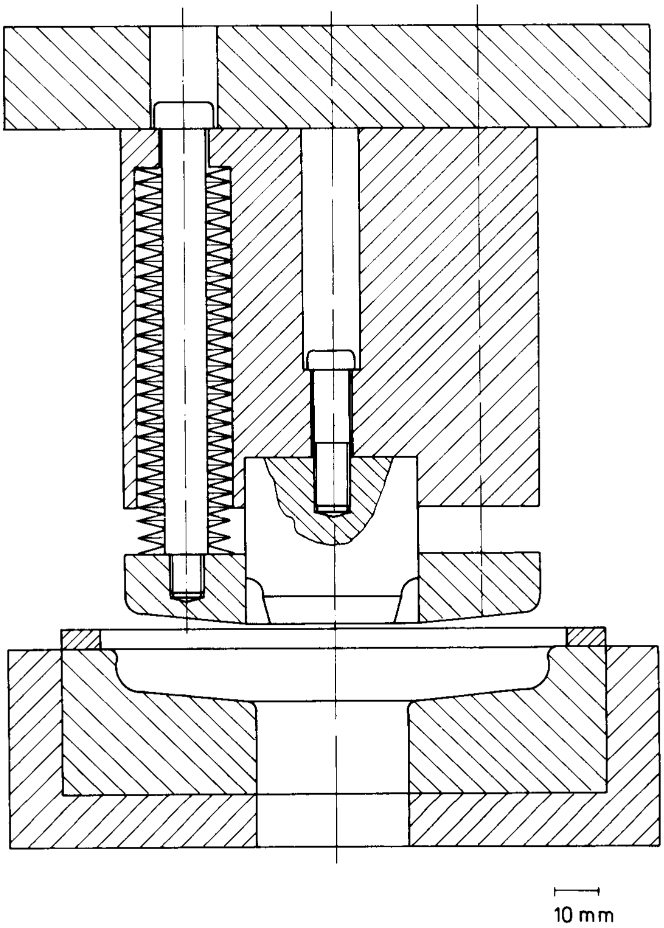


2a)



2c)

Fig. 2. Deep-drawing tools used by Siemens[19] (Table V)



2b)

Table V: Typical deep-drawing procedure [19]

1. Material 3 mm, cross rolled
2. cut circular plates 141 Ø
3. cut central hole 35 Ø
4. anneal at 900°C, 1 h, 10^{-5} torr
5. make mechanically plane
6. machine flat on both sides 2.65 mm thickness!
7. deep-draw 1. tool (outer edge)
8. increase central hole, make central to outer radius
9. deep-draw 2. tool
10. anneal 1200°C, 1 h, 10^{-5} torr
11. deep draw 3. tool
12. machine seam for electron beam welding

3. Other forming techniques

The cylindrical part of the KfK-DORIS-Cavities is simply rolled and longitudinally welded. The diameter tolerance achieved is 0.3 - 0.5 mm.

At HEPL [20] the cavities are shaped by hydroforming and are machined afterwards. SLAC [21] has investigated a special technique called coining. There the exact amount of material is brought between two dies and is squeezed until it fills the entire volume. Removal of the parts requires a heavily oxidized niobium surface.

V. Welding

Niobium can only be heated or melted in vacuum or in a clean inert gas athmosphere to avoid brittleness. Two welding techniques are widely used: Electron beam welding and Tig (tungsten-inert-gas,argon-arc) - welding. Both methods need much experience and many tests for each new geometry, often for each new material are required. In any case welding from inside is to be preferred, if possible. It is difficult to decide which method is superior, the choice is usually made by the availability of the installations existing at the individual place. In table VI a comparison is tried. Fig. 3a-d show some examples of seam shapes used for electron beam welding, the voltage and current values given can only be used as starting points for detailed welding tests. Fig. 4a-c continue this for TIG welded parts. Some parameters for TIG-welding are collected in Table VII.

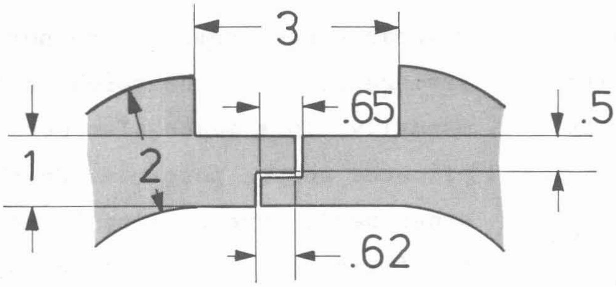
Table VI: Comparison between EBW - and TIG

Electron beam welding	TIG-welding
Requires tight tolerances of the parts to be welded, which are difficult to achieve with large parts formed from sheet. (But: see Fig.3c!)	Tolerance requirements are not so stringent, since the torch is guided manually. This makes also more complicated shapes possible. On the other hand, some tooling is required for fixing the parts during welding.
Complicated shapes of the weld have to be avoided, as all movements in the box must be mechanically and remote controlled.	Although the welds are macroscopically uneven and much broader than with EBW the RF-results are not distinguishable. No spatter has been observed.
Welding from outside often gives rise to uneven inner surfaces. Spattered little Niobium balls often have to be removed mechanically or chemically.	Pumping of the box is also necessary before fitting it with Argon or He. After some welding ($\sim$ about 1 m of weld) the outgassed contaminants have decreased the purity of the argon so much, that an exchange of the argon becomes necessary. Very clean Argon (or He) is absolutely essential.
Rather long waiting times are needed for pumping and cooling.	
The great advantage is the fact, that little heat is brought in and only little deformation by stress relieve can occur. Also delicate parts in the vicinity of the weld remain cooler. (e.g. ceramic windows).	
EBW is considered more expensive, but conclusive numbers are not available.	

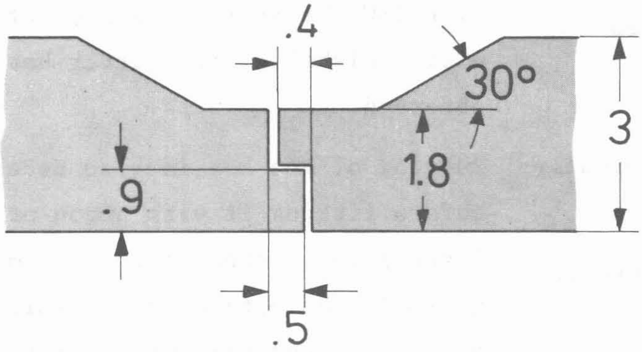
Table VII: Parameters for TIG-welding

Thickness (mm)	Current (A)	Welding speed (cm/min)
.3	40	$\sim$ 50
.5	60	$\sim$ 50
.75	80	$\sim$ 50
2	110	$\sim$ 50

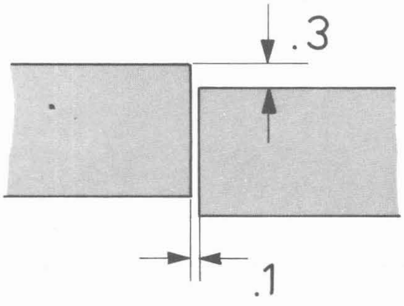
Fig. 3: Examples for Electron Beam Welds



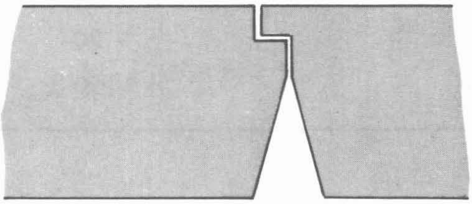
3a:
Interatom-Wuppertal
110 - 130 kV
3.8 - 4.4 mA
shrinkage 0.15 mm



3b:
Siemens (RF-Separator)
110 kV
4.5 - 4.8 mA
shrinkage 0.2 mm

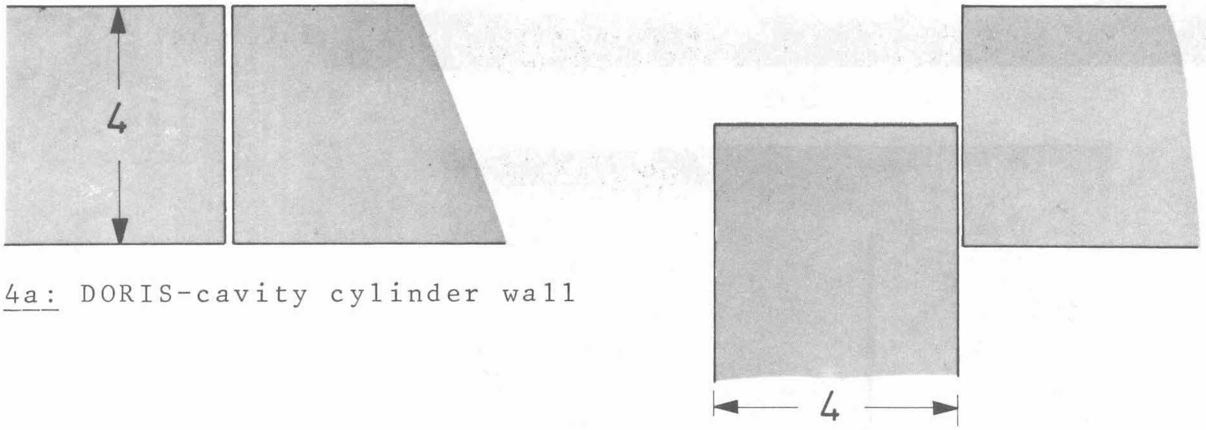


3c:
CERN (500 MHz-cavity)
55 kV
42 mA
shrinkage 0.15 mm



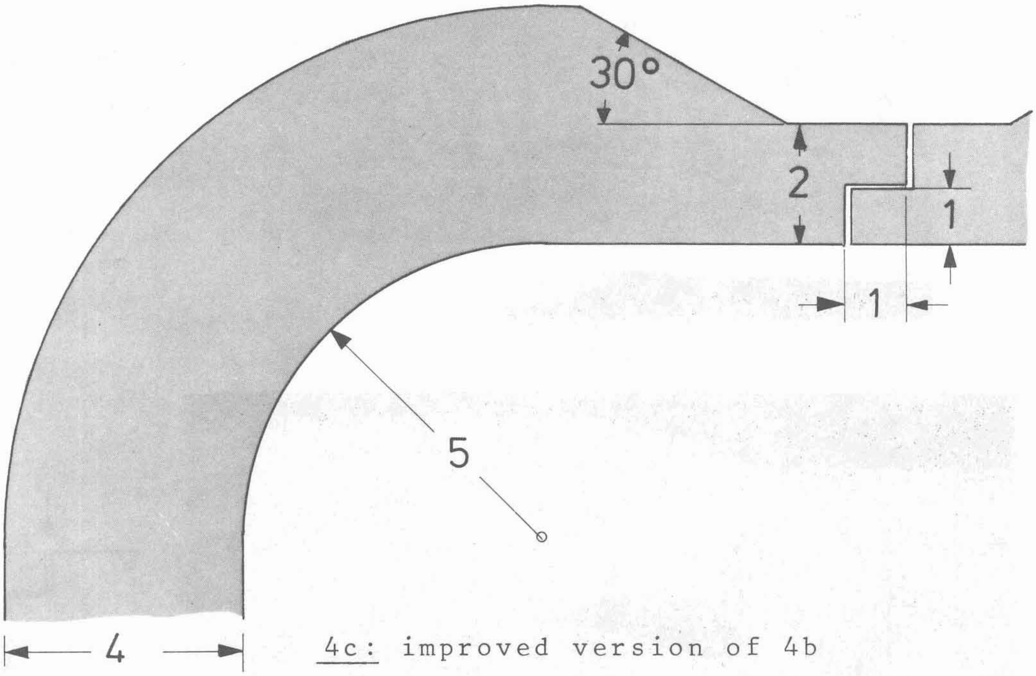
3d:
I. Ben-Zvi
100 kV
20 mA
shrinkage negligible
electron monitor inside!

Fig. 4: Examples for Argon-Arc-welds



4a: DORIS-cavity cylinder wall

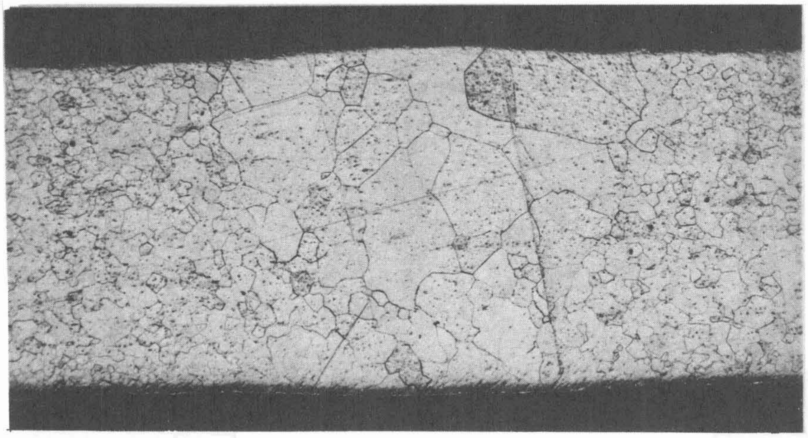
4b: DORIS-cavity endplate - cylinder wall



4c: improved version of 4b

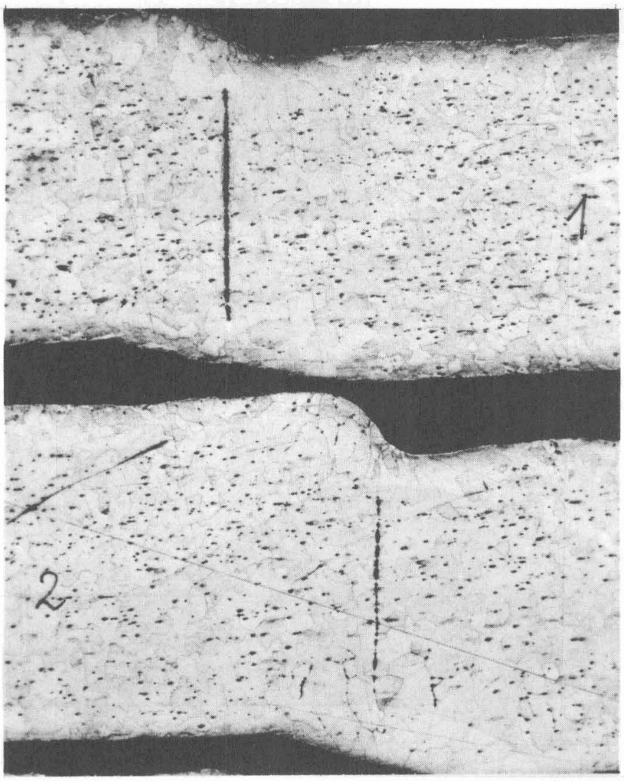
Fig. 5:

EBW-tests Interatom



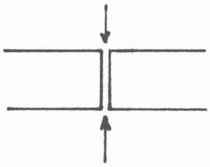
1 single piece, 1 mm
beam from both sides
grain size!

5 a

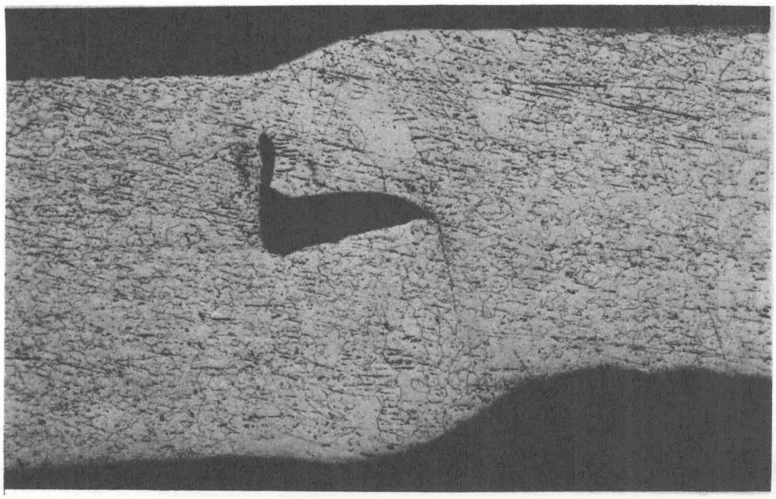


5 b

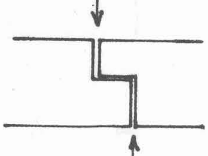
2 pieces 1mm



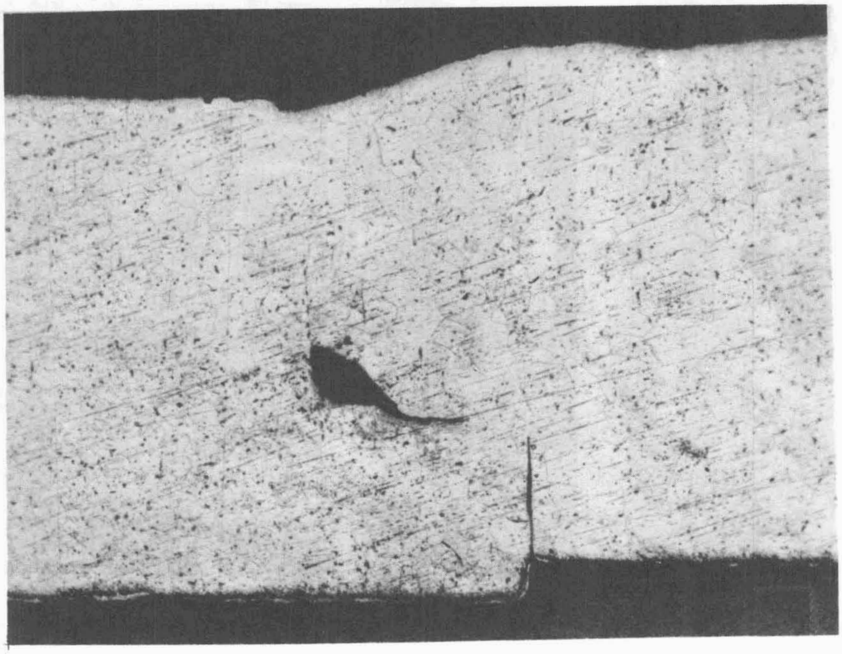
remaining slot,
shift!



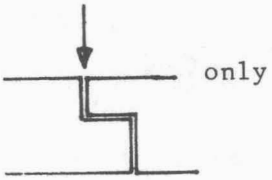
5 c



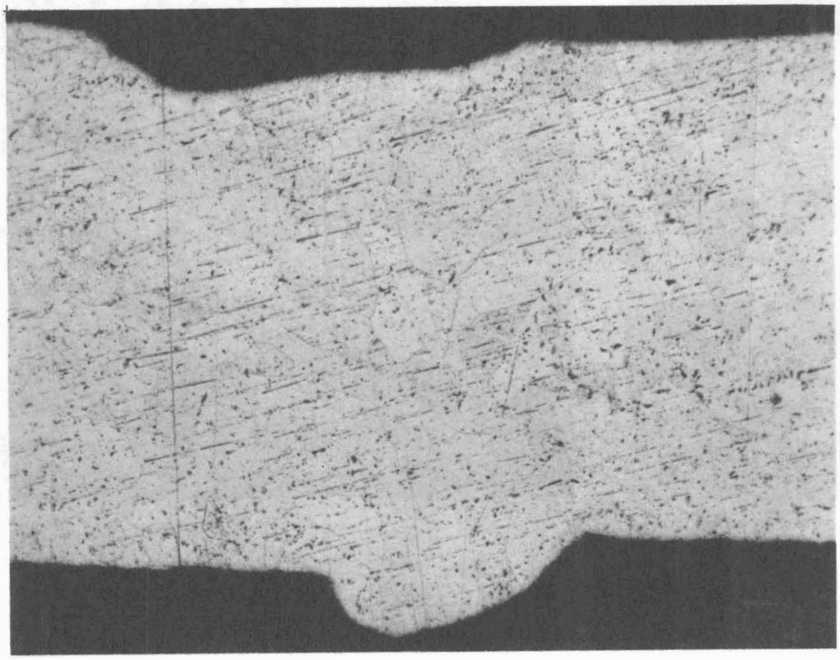
remaining hole
shift!



5 d



remaining hole and slit,
shift



5 e

same as above,

more current

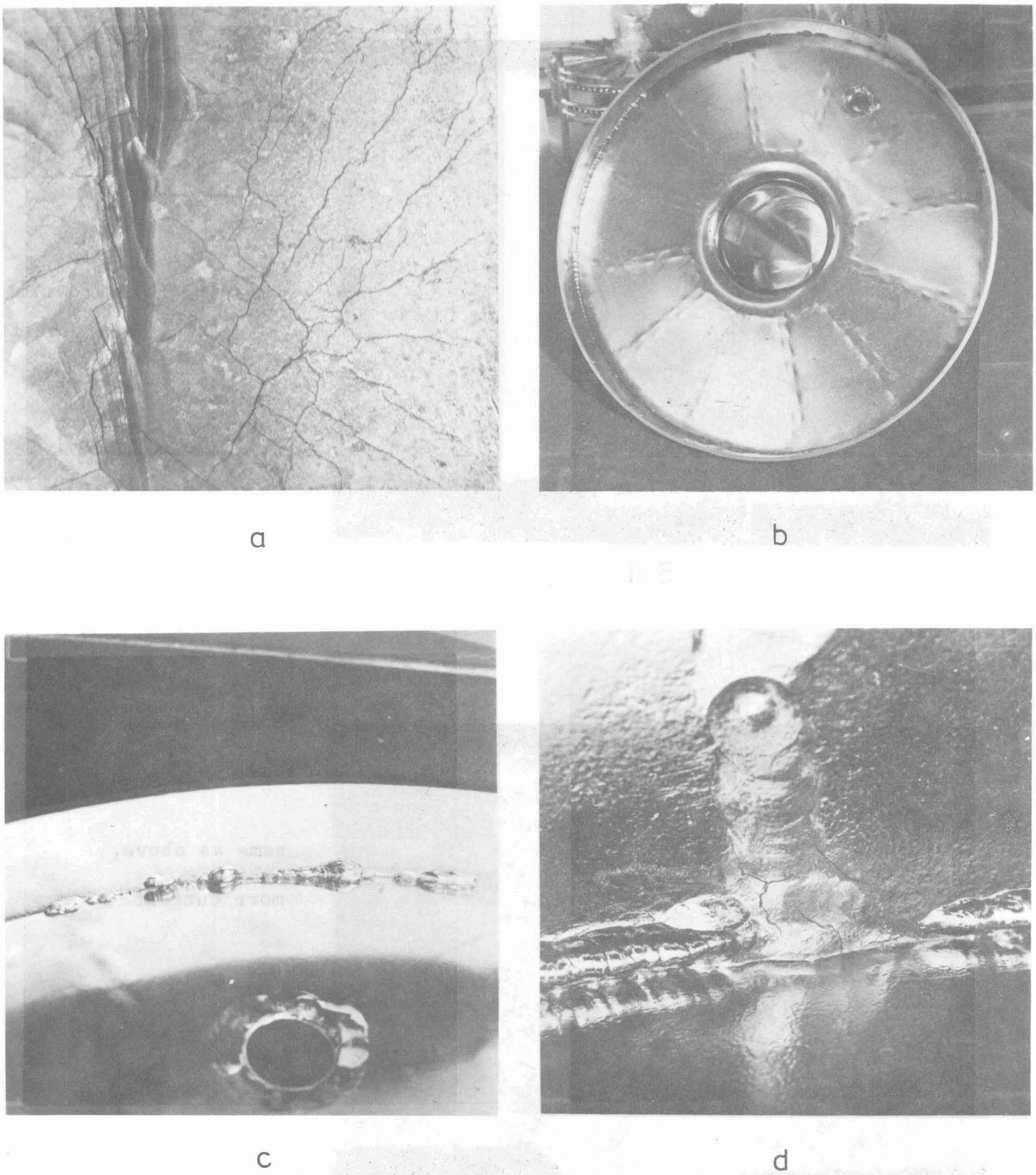


Fig. 6: TIG-welding the DORIS-cavities

a: fissures in cylinder wall
b: endplate - cylinder wall
c: Detail of b
d: crossing of welds in cylinder wall and corner

Fig. 5a-e show a few examples of electron beam welding tests made by Interatom.[22]

The fotographs Fig. 6a-d taken during the fabrication of the DORIS-cavities show that argon-arc welding sometimes gives rise to difficulties: Fissures in the longitudinal weld of the cylinder wall originating in the weld and extending into the material next to the weld could be explained by a not completely clean box (Siemens). Similar cracks together with a mat surface of the weld were observed at Heraeus, which were explained by some mysterious behaviour of the material that did not show up by any chemical or metallurgical analysis. This problem could be overcome by heating the material to 1800°C in a vacuum of 10^{-8} torr. All welds done after this heat treatment appeared shiny and vacuum tight. The weld in the corner between endplate and cylinder wall of DORIS I shown in Fig. 6c-d was very uneven, especially at the crossing between the longitudinal weld in the cylinderwall and the corner weld. It was rewelded from inside at KfK. The two DORIS-cavities made by TIG-welding were 1-3 mm out of tolerances. Apparently more effort is needed to fix the parts in the box during welding, although not all tolerance problems can be attributed only to the welding.

CERN[23] reported about TIG-welding without a box, where a stream of argon is guided along the weld in such a way, that no air contaminates hot parts.

Another welding method has been described by Isagawa[24], and was also used at KfK for welding niobium tubes on sheet [25]: Clean, smooth niobium surfaces are mounted in an ultrahigh vacuum furnace and pressed by niobium weights. By diffussion at 1800°C the parts are tightly welded. This method certainly avoids contamination better than any other and combines a heat treatment which is considered desirable for other reasons with the welding. For large cavities made of sheet, however, it might be difficult to apply the necessary weight.

VI Costs

It is impossible at the moment to answer the question about large scale fabrication costs precisely. Experience exists only for some single cavities and very preliminary quotations have been obtained for the production of ∿ 1600 m of cavities in the frequency range 350-700 MHz. More conclusive numbers can only be obtained after a real design has been made, which certainly has to take into accont minimizing the fabrication costs. Since the material costs have been increased considerably during the last year, it seems advisable, to reconsider niobium-saving fabrication methods like sputtering or electroplating thin layers of Nb or explosion-bond [2] thin Nb-sheet on copper. This, of course has to take into accont the questions of heat transfer [4] and the pro's and con's of the heat treatment at high temperatures. By electroplating one may increase the Nb purity with positive consequences for heat transfer.

In addition to the fabrication and material costs one has to take into account the costs for surface treatments which generally are dominated by manpower and installation costs. (One surface treatment of the DORIS cavity requires DM 200,-- for chemical solutions + 2 man-days + an existing installation that has costed about DM 100.000,-- + 4 days treatment in a furnace that has costed 1 Million DM!)

Table VIII collects some figures of the amounts involved which should show the direction, where to go to arrive at reasonable fabrication costs.

Table VIII: Costs

1. Single cavities	fabrication KDM	material KDM	total KDM
DORIS I/II (TIG, 4mm wall)	20	18	38
DORIS III (offer: EBW)	30 (+ 10 tools)	18	48 (58)
CERN 500 MHz (EBW, 2 mm)	16	7.2	23.2
2. Mass production	fabrication KDM/m	material KDM/m	total KDM/m
Interatom 3 GHz	27	∿ 3.5	30.5
Cornell[26] 1,5 GHz			30*
SLAC[26] 1.5 GHz			54*
Inquiries 1600 m:	KDM/m	KDM/m	KDM/m
Single cells 350 MHz	12-30	30	42-60
Single cells 500 MHz	14-31	25	39-56
7-cell-Iris 700 MHz	7- 9	17	24-26

* including couplers,tuners ...

VII. Conclusions

The fabrication methods for niobium cavities have been reviewed. Machining, sheet metal forming and welding techniques have been described. Handling of niobium presents no particular difficulties, but it requires much experience, The transfer of the know-how gained in the laboratories to the industrial scale is possible, but needs some training. It cannot be repeated often enogh that niobium is a very soft material ,and that therefore all inner surfaces and especially all flanges have to be protected against accidental scratches. To reduce material costs methods of producing thin Niobium layers on a copper substrat should be investigated again.

Acknowledgements

I would like to thank all laboratories and companies, who have contributed to the informations collected in this review, especially CERN, Cornell, HEPL, SLAC, the University of Wuppertal and the companies Interatom, Kawecki, Siemens, and Wah Chang. The state of the art reported is the outcome of more than 10 years of competent work of many technitians in all laboratories and companies where niobium cavities have been built. Their effort should be greatly acknowledged.

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