Materials for the Flanges of TESLA Cavity

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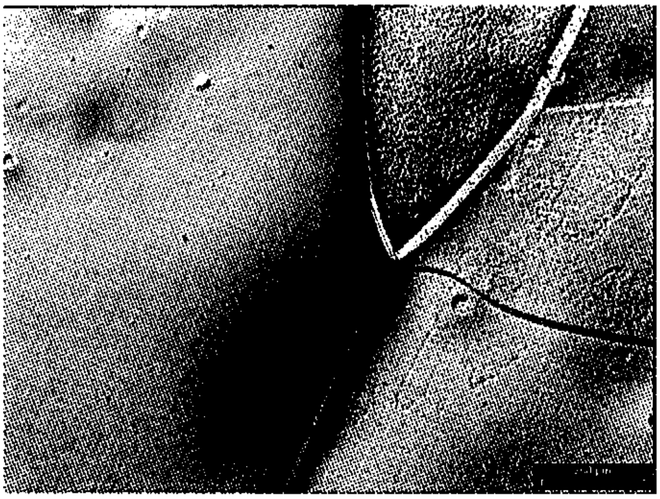
First experiences with TTF flanges of Nb40 have shown, that after 1400°C annealing the flanges become too soft and get rather large grain size. This is probably the reason for some leakage problems with the helicoflex seals. Further development both of the flange material and of the seal system is necessary. The metallurgical properties of some materials suitable for this aim are investigated and compared below.
The material completely suitable for the flange connection should have following properties.

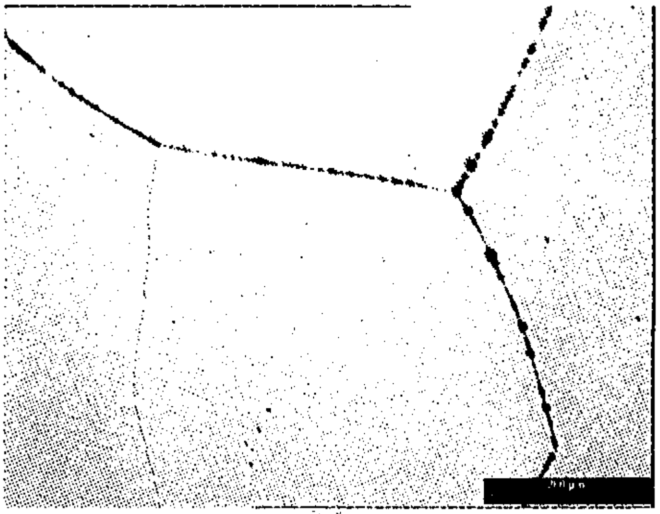
- welding connection with Nb without formation of brittle phase should be possible.
- should be significantly harder and stronger than pure Nb. This reduces the risk to damage the sealing surface and increases the number of choices of sealing systems. As small as possible grain size after solid state gettering with Ti at 1400°C for 4h is very desirable for sealing function.
- The machining of the material should be possible.
- The corrosion rate under chemical etching should be as small as possible, at least comparable with pure Nb. Protective actions can be omitted in this case.
- The cost of the material should be comparable with Nb40 (RRR=40).

We analyzed three types of electron beam melted materials, that some companies produce routinely, and which can be easily purchased at the moment:- NbZr1%, TaW2,5%, NbTi50%. The states of as received and annealed at 1400°C material were examined. Niobium and NbZr1% were delivered in the annealed at 800°C condition.
The Vickers hardness HV, tensile strength Rm, yield strength Rp0.2, breaking elongation A, etching rate and grain structure were tested.
The hardness was measured with the SHIMADZU HMV-2000 device. The tensile tests were done at a strain rate 10^{-3} s^{-1}. The sample preparation for the microstructure analysis was done mechanically with subsequent etching in the acid mixture HF(40%)/HNO_3(65%)/H_3PO_4(85%) in volume ratio (1:1:2). The corrosion rate was examined in the same acid mixture at room temperature. The samples with dimensions 50x20x3 mm were immersed into the acid for 200 minutes.
Some results of the investigations are summarized in the table 1. The microstructure pictures of Nb, NbZr1%, TaW2,5% and NbTi50% after annealing at 1400°C, 4h are shown in figure 1. It can be clearly seen, that both NbZr1% and TaW2,5% demonstrate a completely recrystallysed microstructure with a rather small grain (ASTM grain size 4-6). By contrast the at 1400°C annealed NbTi50% as well as pure Nb show significant grain growth, so that the grain size reaches a few mm.



a

b



c.

d

Fig. 1 Microstructure of Nb (a), NbZr1% (b), NbTi50%(c), TaW2,5%(d) all annealed at1400° C

Table 1. Comparison of the metallurgical properties of the flange materials with those of pure Nb

as received

properties	Nb300, (800°C annealed)	NbZr1%, (800°C annealed)	TaW2,5%	NbTi50%
yield strength Rp0,2, N/mm2	50-70	265	675	
tensile strength Rm, N/mm2	150-180	320	685	370
breaking elongation A, %	30-40	6,5	4	26
hardness, HV0,05	50-60	125	418	150
grain size, µm	50-100	not recrystal.	not recrystal.	not compl.recr.
etching rate, µm/min	1	1,5	0,35	1,63

annealed 1400°C, 4h

properties	Nb 300	NbZr1%	TaW2,5%	NbTi50%
yield strength Rp0,2, N/mm2	30-40	140	330/290	
tensile strength Rm, N/mm2	110	320	427	500
breaking elongation A, %	26	28	39	4,5
hardness, HV0,05	50	110	121	182
grain size, µm	1000-3000	50-150	30-100	2000-4000
etching rate, µm/min	0,99	1,48	0,33	1,25

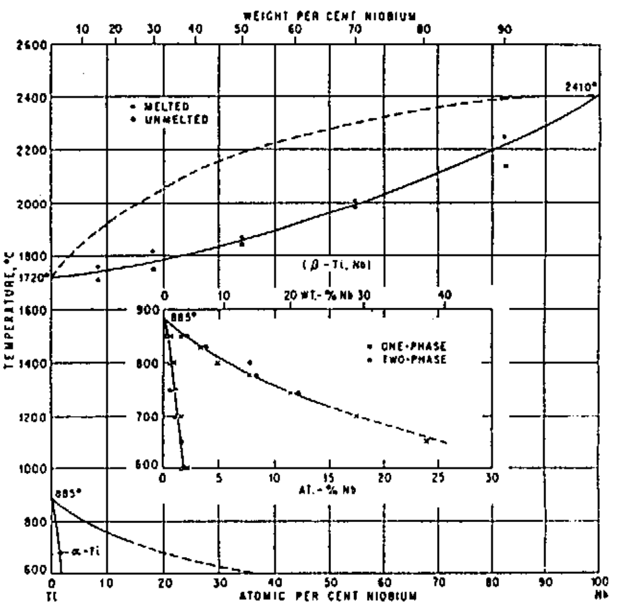


Fig.2 Phase diagram of Nb-Ti system

The microstructure pictures allow to conclude, that the annealing temperature1400°C is close to the recrystallization temperature of NbZr1% and TaW2,5% alloys. The recrystallization is already completed, but the grain growth is not significant. On the contrary the 1400°C annealing temperature substantially exceeds the recrystallization temperature of NbTi50% and pure Nb and caused the observed grain growth.
It is well known, that the recrystallization temperature of the metals is strongly dependent on the purity and the degree of cold deformation. But roughly, the recrystallization temperature can be estimated as 1/3 of the melting temperature T_{melt}. Additions of 50%Ti to Nb reduces the melting point and accordingly the recrystallization temperature. This can be clearly seen on the binary phase diagram of NbTi alloys (figure 2).
Dissolving of Zr in Nb does not change the melting temperature significantly /1/. In this case it is important, that the grain growth in substitution alloys is influenced by the relative lattice parameter change $\Delta a/a$ (a_{Nb}=3,301 A°, a_{Zr}=3,609 A°). The difference in atomic size determines the degree of grain boundary pinning /2/. So that already small addition of Zr to Nb increases the recrystallization temperature from about 800-900°C of Nb /3/ to about1300-1400°C. Tungsten creates with Ta a continuous series of solid solutions /1/. Dissolving of W in Ta increases the melting point and the already rather high recrystallization temperature of pure Ta (1000-1100°C) /2/. The difference in the atom sizes of these two metals (a_{Ta}=3,296 A°, a_W=3,158 A°)is not so large.
It is important to be sure, that no brittle phases (intermediate compounds, segregation of the second component and so on) appear at the welding connection area, which can influence the ductility. In order to check this, the electron beam welding of Nb with each potential flange material NbZr1%, TaW2,5% or NbTi50% was done and the welding quality was tested.

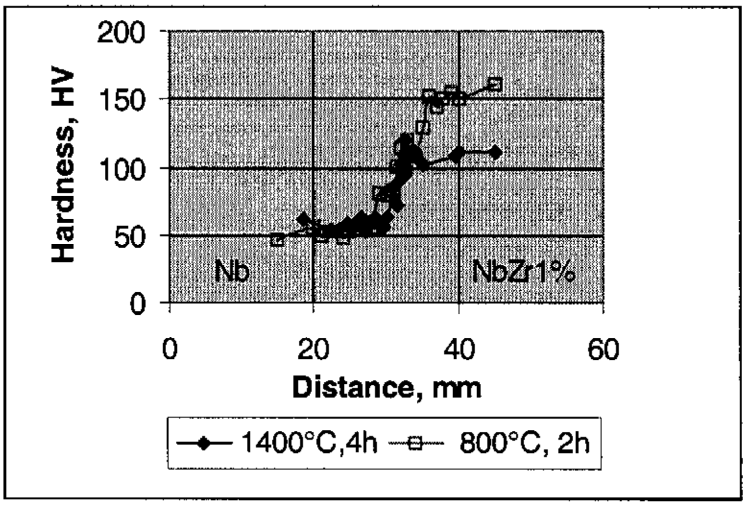


Fig.3. Hardness HV0.05 in the area of the welding connection Nb-NbZr1%

The behavior of the hardness in the welding area of Nb-NbZr1%- and Nb-NbTi50%-connection can be seen in figure 3 and figure 4. The hardness of Nb-NbZr1% changes smoothly from the value of the pure Nb to the HV value of the alloy NbZr1%. A similar hardness behavior in the welding area was obtained for TaW2,5% alloy (figure 5). The tensile test confirms, that an EB welding of these materials is uncritical. For example the behavior of the strain–stress characteristic of the welding connection Nb-NbZr1% after annealing at 1400°C is compared with correspondingly annealed Nb and NbZr1% alloy (figure 6). The breaking elongation is reduced, but nevertheless is acceptable. The tensile strength and especially the yield strength remain rather high. Absence of problems in the welding connection of Nb-NbZr1% and Nb-TaW2,5% can be expected from the binary phase diagrams of Nb-Ta; Nb-W and Nb-Zr. The components create a substitution body-centered cubic (bcc) solid solution in the analyzed concentration ranges /1/.

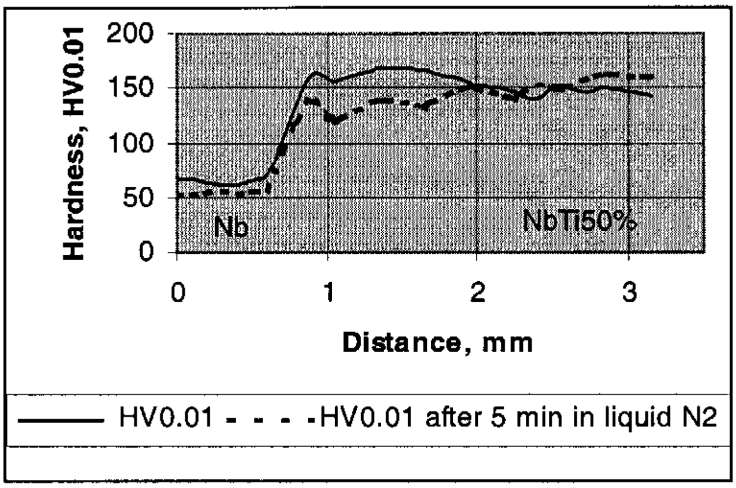


Fig.4. Hardness HV0.1 in the area of the welding connection Nb-NbTi50%

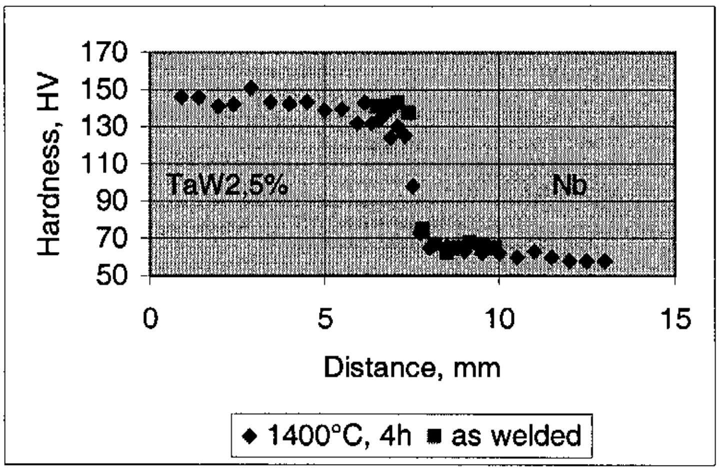


Fig.5. Hardness HV0.05 in the area of the welding connection Nb-TaW2,5%

The situation is much more complex for the NbTi alloys. As it can be seen from the binary phase diagram, a hexagonal α-phase appears at the Ti side. A wide transition region from α to β-solid solution exists. Moreover a martensitic type of transformation $\beta \Rightarrow \alpha'$ near to the for us interesting composition of NbTi50% is likely at room and low temperatures. The critical concentration of Ti at low temperatures unfortunately is not well known. For example, like /3/, the HV of the alloy NbTi60% changes from 150 till 260 after fast cool down to temperature of liquid nitrogen and

Table 2. Comparison of the advantages and disadvantages of the materials for the cavity flanges

NbZr1%		TaW2.5%		NbTi50%	
advantage	disadvantage	advantage	disadvantage	advantage	disadvantage
mechanical strength is much higher, than of Nb		mechanical strength is much higher, than of Nb		mechanical strength is much higher, than of Nb	
high hardness, even after annealing at 1400°C		high hardness, even after annealing at 1400°C		high hardness, especially after annealing at 1400°C	large grain after annealing at 1400°C
small grain, even after annealing at 1400°C		small grain, even after annealing at 1400°C			danger of the second phase forming
acceptable EB welding connection		acceptable EB welding connection		acceptable EB welding connection	
acceptable etching rate		small etching rate		acceptable etching rate	etching can be critical for the sealing because of the large grain
machining is possible		machining is possible		machining is possible	
costs are comparable with Nb40 (120 $/kg)			costs are rather high (200 $/kg)	costs are comparable with Nb40 (100 $/kg)	
density is comparable with Nb (8,6 g/cm3)		density is rather high (16,6 g/cm3)	heavy for handling	density is less than of Nb (6,5 g/cm3)	

subsequent annealing at 425°C, 40h. However the hardness of the alloy NbTi50% remains stable after the same procedure. Traces of the second phase can be seen in some grain boundaries of NbTi50% alloy after annealing at 1400°C and slow cool down (figure 1c). All this means is, that instability of the properties of NbTi50% may

be observed, especially at the welding connection, if the part would be exposed to radical temperature variation. Therefore it was reasonable to check the hardness of the alloy NbTi50% after cool down to low temperatures.
The hardness distribution in the EB welding region of Nb with NbTi50% as welded and after positioning for 5 min in the liquid nitrogen can be seen in the figure 4. The hardness alteration is rather smooth. No increase of the hardness was detected after cool down in liquid nitrogen.

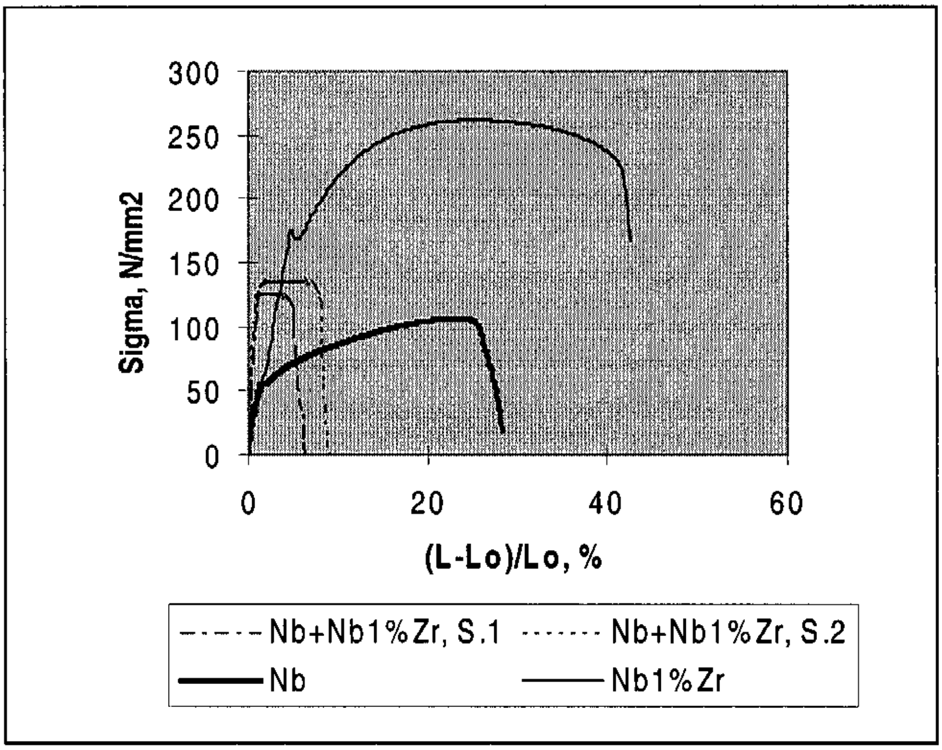


Figure 6. Comparison of the strain–stress curve of the annealed at 1400°C, 4h Niobium, NbZr1% alloy and its welding connection

The demonstrated results allows to make a comparison of the advantages and of the disadvantages of potential flange materials (Table 2). It can be seen, that all three materials in principle can be used as a flange material for TESLA cavities. Nevertheless the minimal number of the disadvantages is seen for the NbZr1% alloy. A combination of mechanical strength, good machining properties, fine grain after 1400°C treatment and acceptable costs prove it to be a very promising material for the flange fabrication. The production of some flanges from this material and a subsequent testing in UHV at as received and after 1400°C annealing conditions can be strongly recommend as a next step of the investigations.

Acknowledgments

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