Conceivable Bottlenecks of RRR300 Nb Production for ILC Project

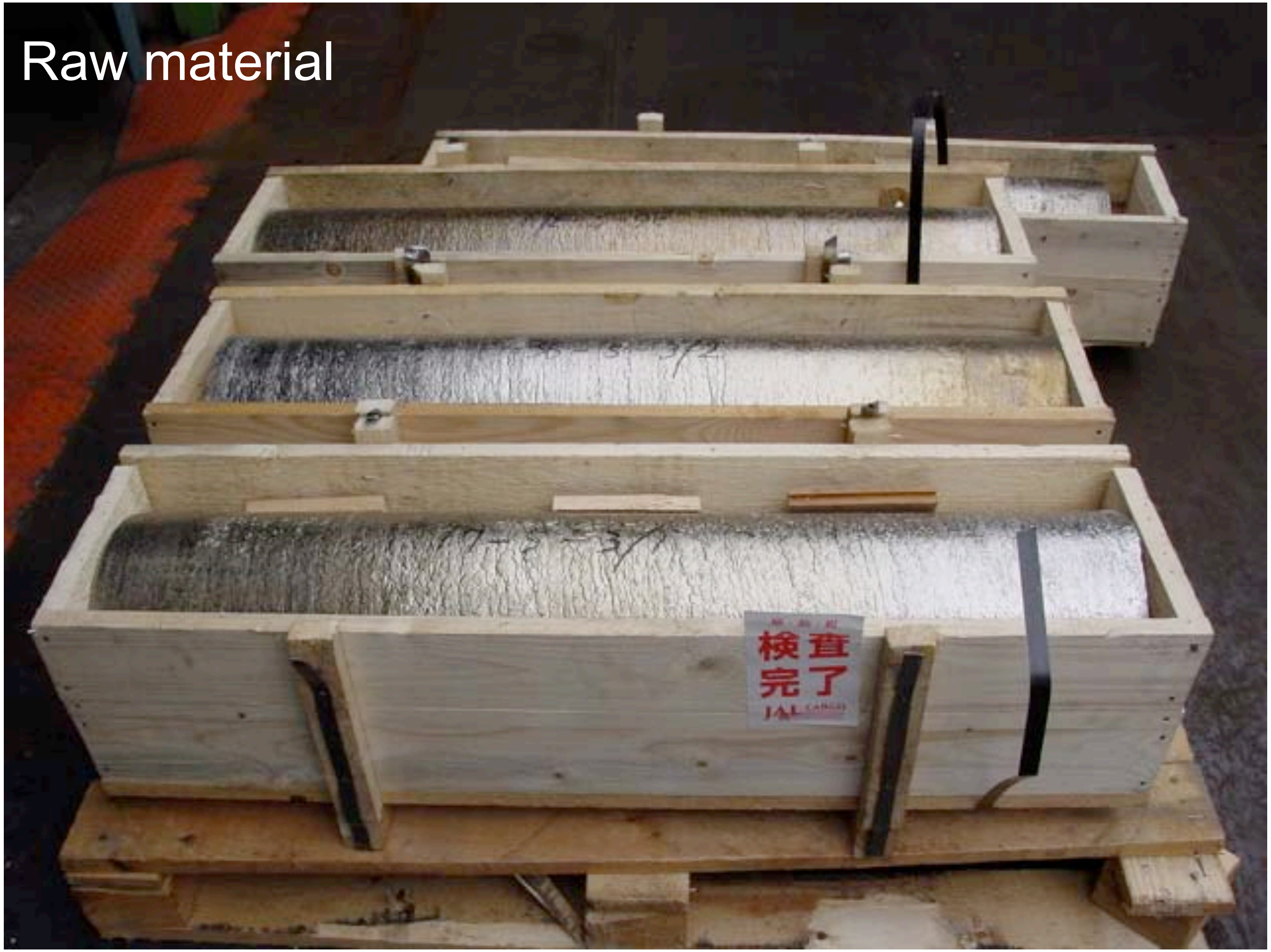
Hiroaki Umezawa

Tokyo Denkai Co., Ltd.

Agenda

1. How to produce high purity niobium.
2. Conceivable bottlenecks of production for huge amount of high purity Nb.

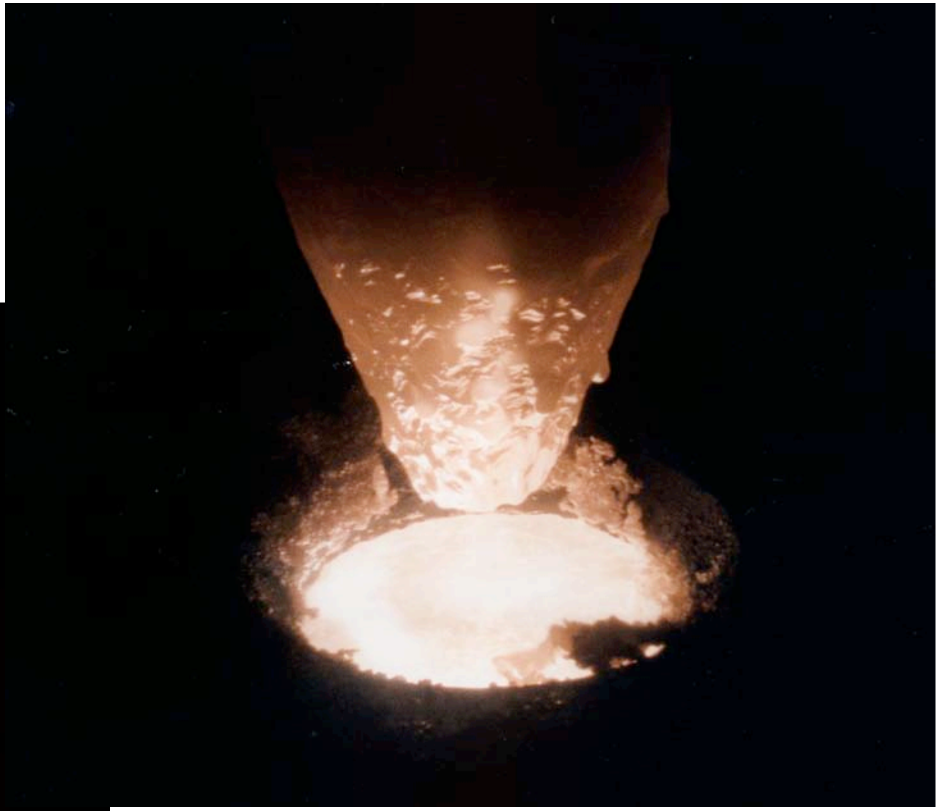
Niobium Ingots



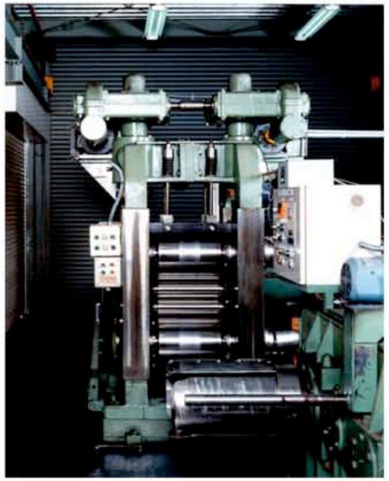
Electron Beam Melting Furnace

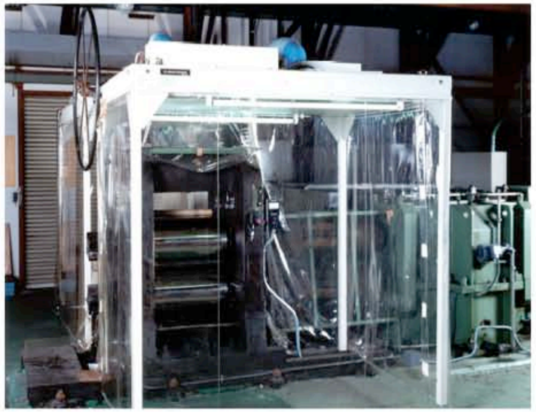
Tokyo Denkai

EB Melting



Rolling mills

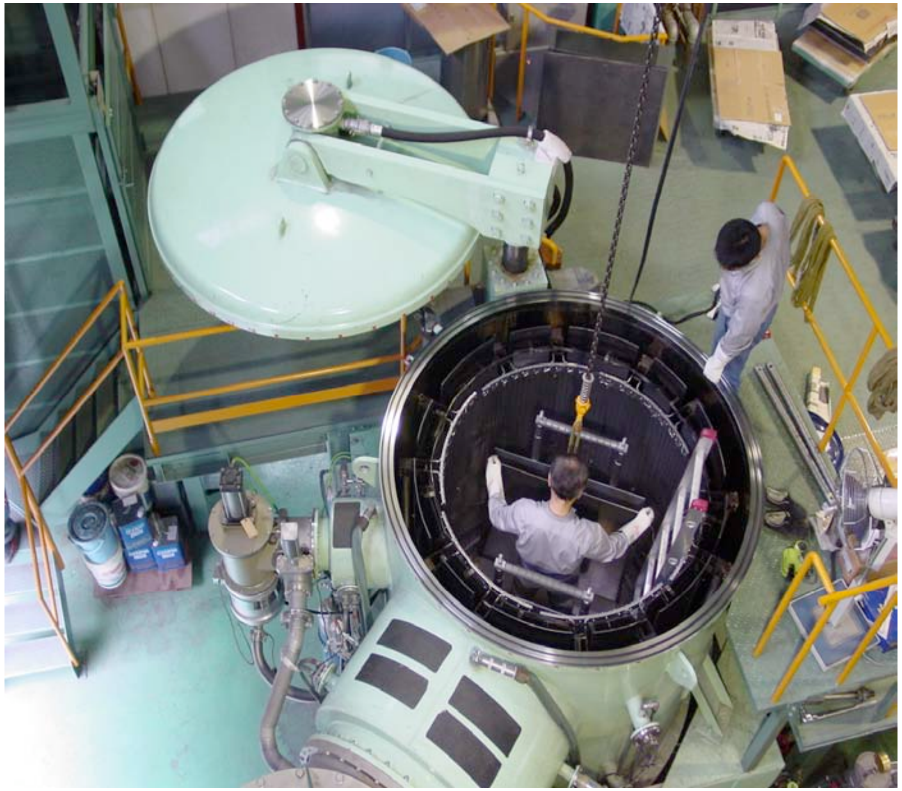




Name	Roll Size		Motor	Specific Sheet Size	
	Diameter	Width		Thickness	Width
Two High Rougher Mill	500 mm ø	1,000 mm	300 kW	60–8 mm	
Two High Finishing Mill	500 mm ø	1,100 mm		8–0.8 mm	900 mm
Two High Rougher Mill	460 mm ø	800 mm	75 kW	8–3 mm	
Two High Finishing Mill	420 mm ø	800 mm		3–0.1 mm	500 mm
Four High Mill	390/130 mm ø	450 mm	37 kW	1–0.1 mm	350 mm
Four High Mill	300/145 mm ø	300 mm	37 kW	0.3–0.08 mm	200 mm
Six High Mill	40/105 mm ø	150 mm	5.5 kW	0.3–0.05 mm	100 mm

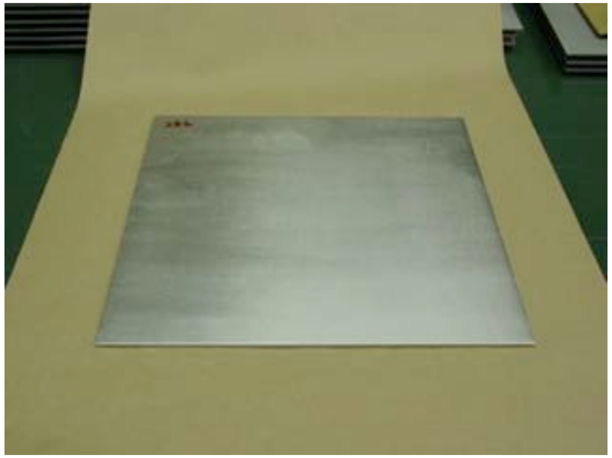
Vacuum heat treatment

Tokyo Denkai



Maximum temperature : 1400 [ºC]
Achievable pressure : 2 x 10^{-5} [Pa]

Niobium sheets



Nb sheets
used for producing
Superconducting cavity material
at
Fermi Lab SMTF

Demand of high purity Nb for ILC project

Superconducting RF cavity for ILC

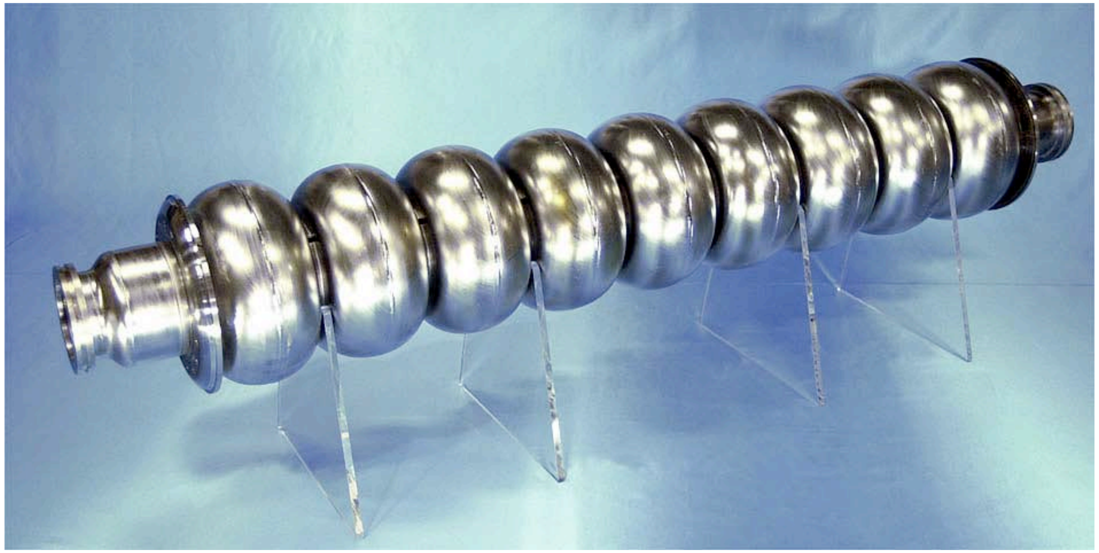
17,000 nine cell cavities

17,000 x 18 half-cells = 306,000 sheets

Nb weight of a sheet for half-cell : 2.8 x 265 x 265 mm = 1,685 g

306,000 x 1.685 kg/sheet = 515.6 ton

Backup cavity, beam tube, HOM coupler, etc…about 600 ton

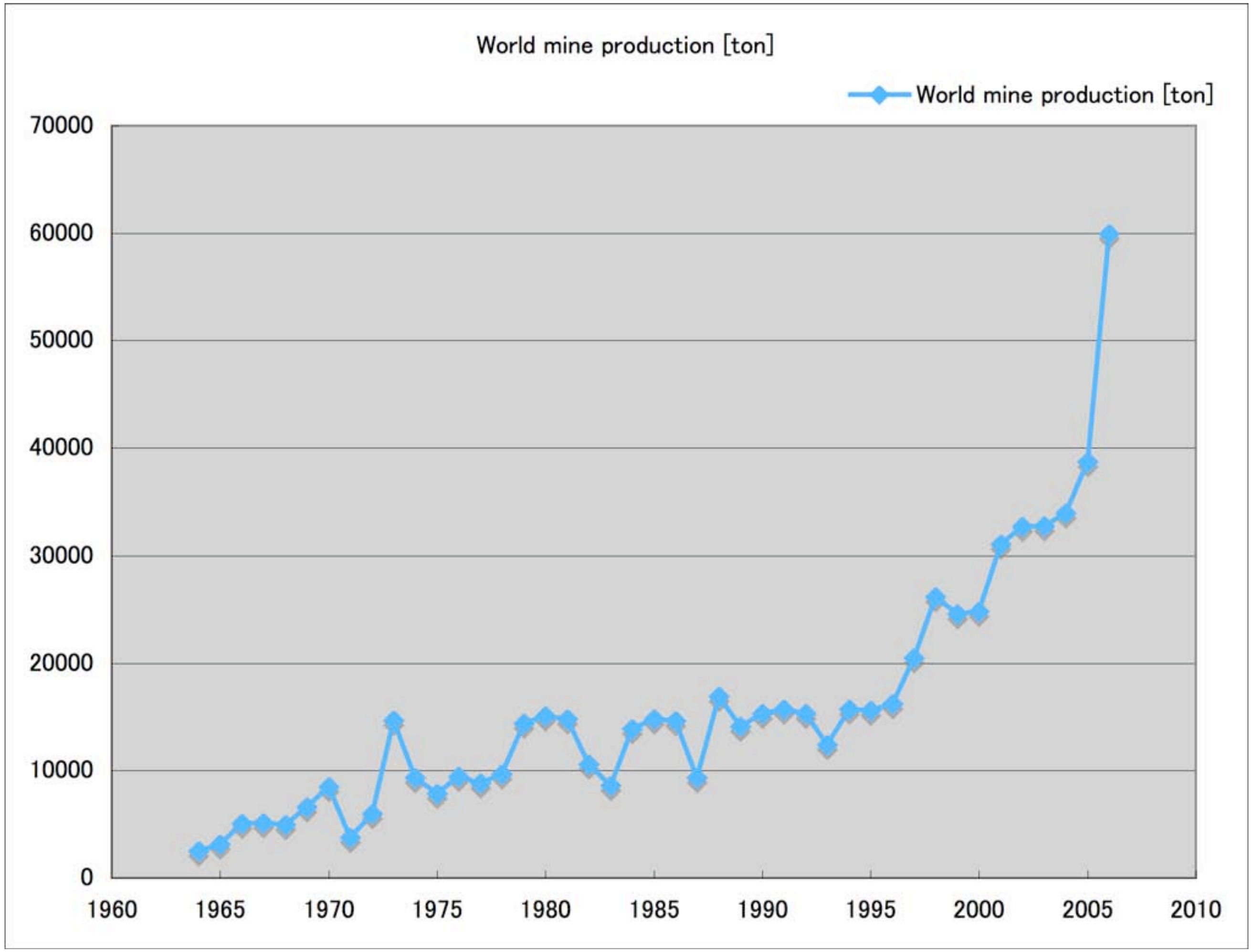


http://lcdev.kek.jp/photos/20050513/20050513IchiCav.jpg

ILC 9-cell Cavity made with high purity Nb
17,000 cavities / 3 years

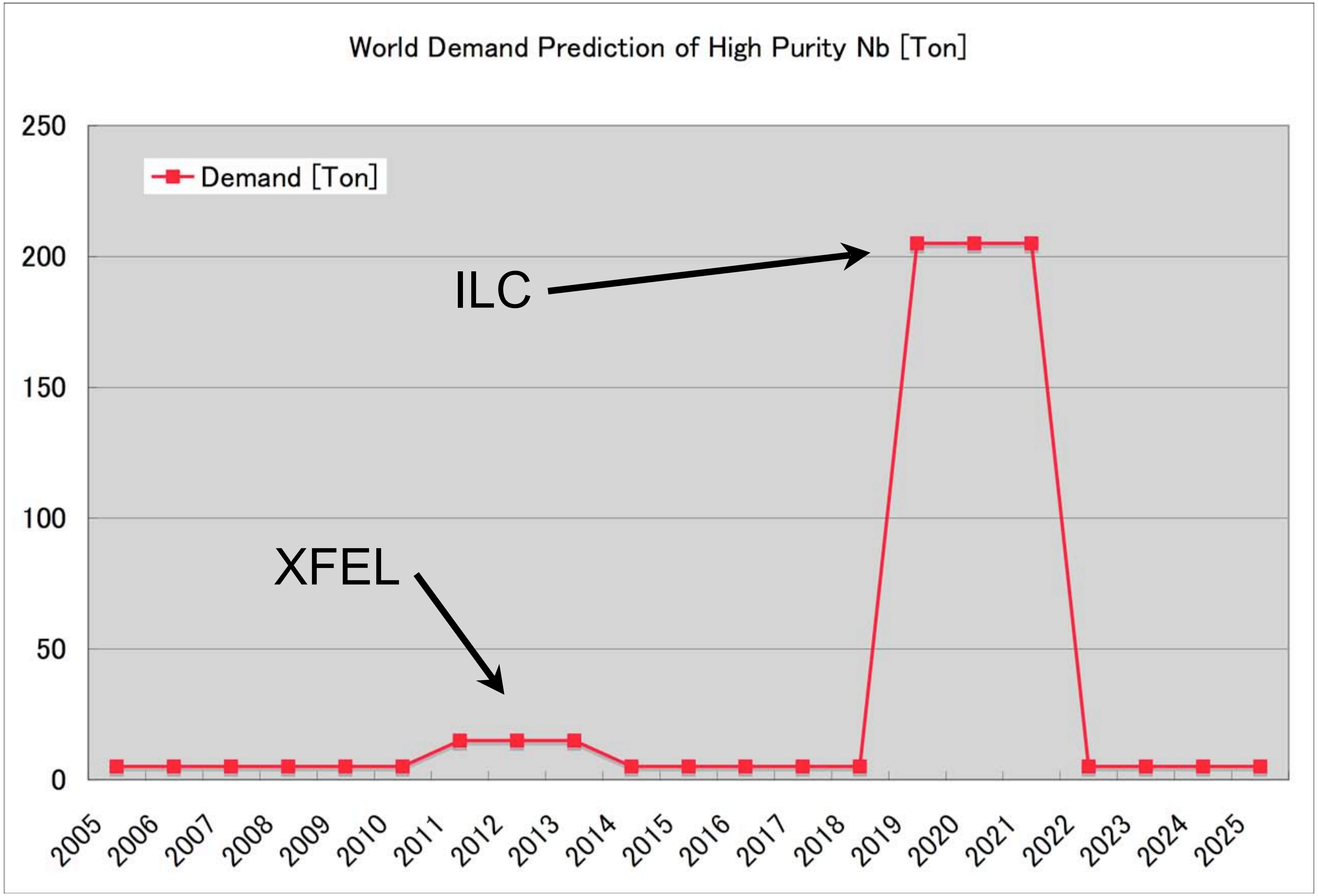
Demand of High purity Nb is 600 ton in 3 years.

Niobium world mine production



U.S. Geological Survey, Mineral Commodiity Summaries 1996−2007

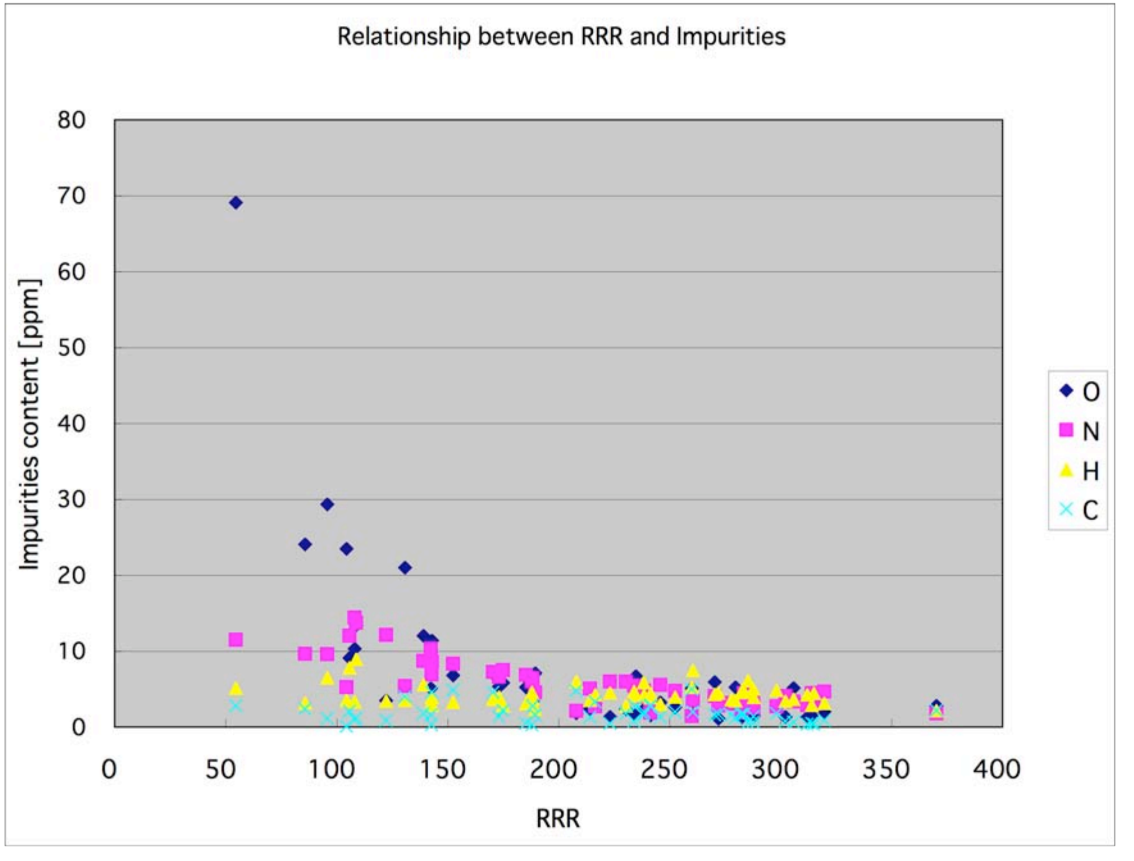
Demand Forecast



Bottleneck 1

- Low Ta concentrate Nb Raw materials

Relationship between RRR and Interstitial impurities



Regression Analysis Result

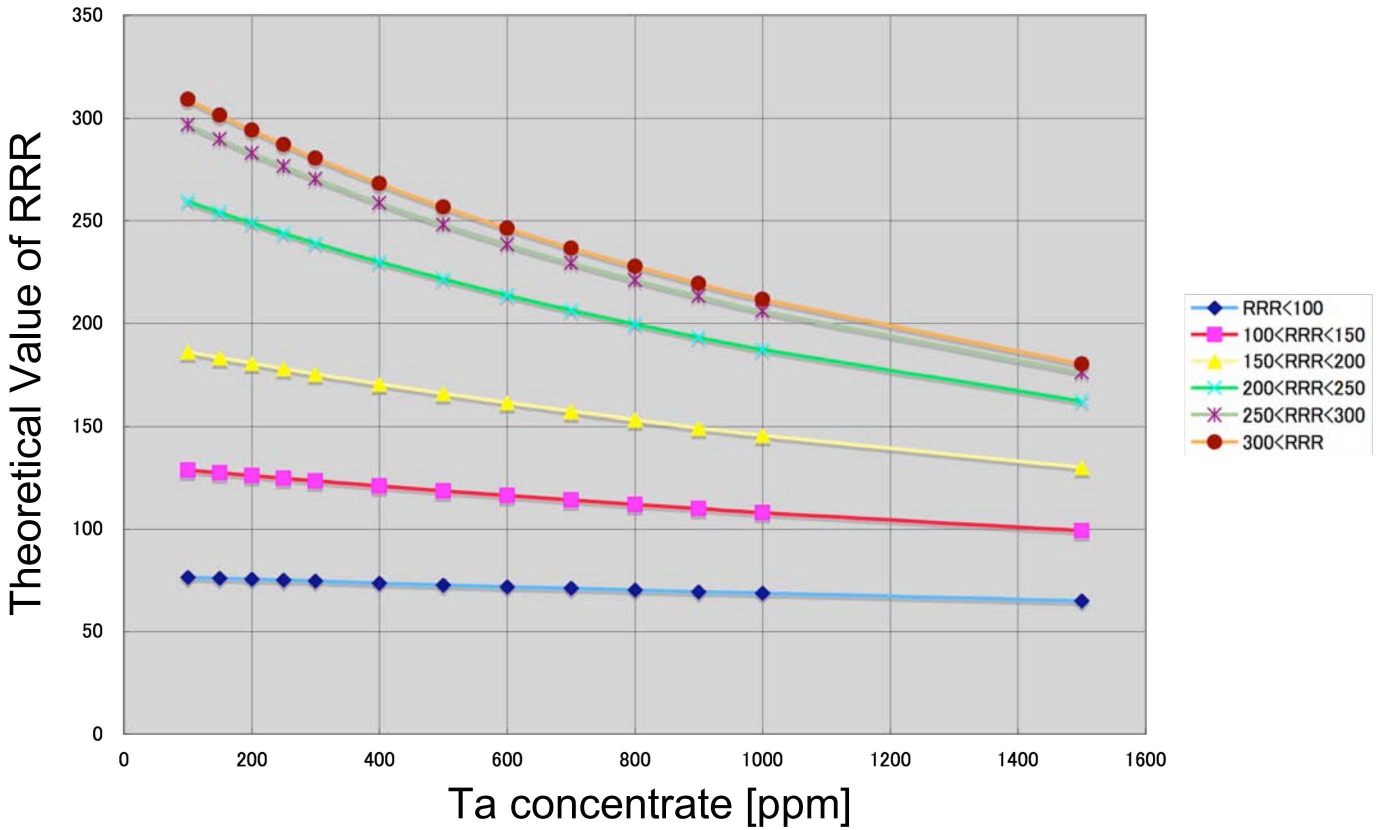
K.K.Schulze: J. Metals, 33(1981), 33-41

$$\frac{1}{RRR} = \frac{O}{5000} + \frac{N}{3900} + \frac{H}{1550} + \frac{C}{4100} + \frac{Ta}{550000} + \ldots$$

Umezawa's calculation.

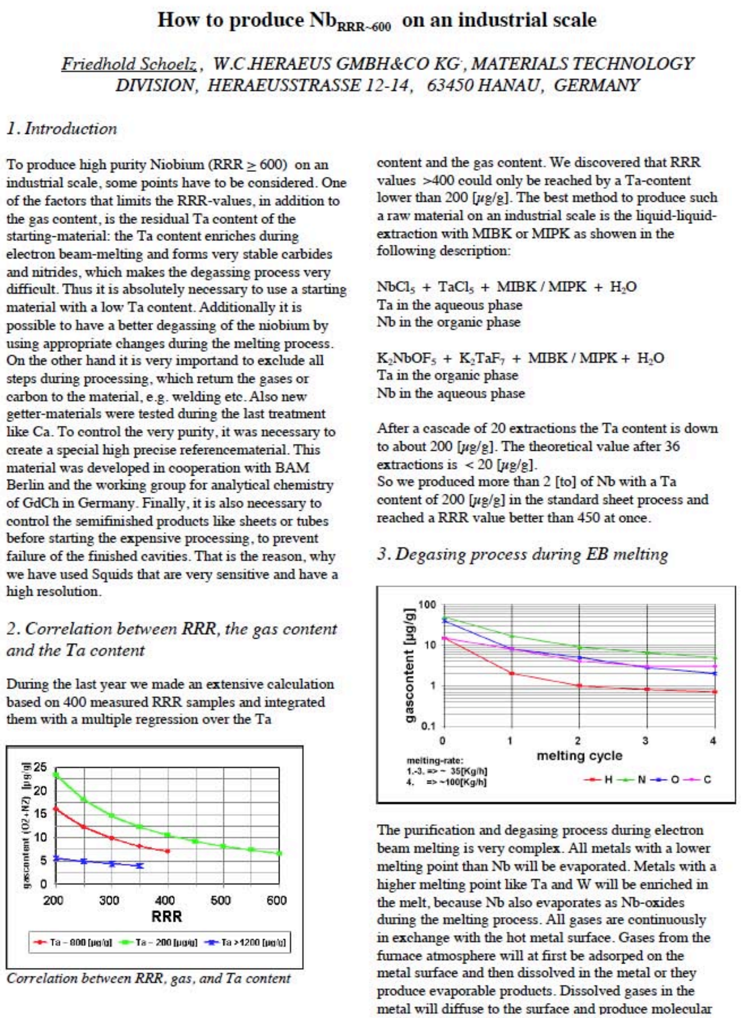
$$\frac{1}{RRR} = \frac{O}{5800} + \frac{N}{2273} + \frac{H}{16322} + \frac{C}{8911} + \frac{Ta}{604690} + \frac{1}{1249}$$

Simulation of Ta influence



$$\frac{1}{RRR} = \frac{O}{5800} + \frac{N}{2273} + \frac{H}{16322} + \frac{C}{8911} + \frac{Ta}{604690} + \frac{1}{1249}$$

F. Schoelz et al., “How to produce NbRRR~600 on an industrial scale”Proc. of the 9th Workshop on RF Superconductivity, Santa Fe, USA, Nov. 1-5, 1999, pp. 100-103



How to produce $Nb_{RRR\sim600}$ on an industrial scale

Friedhold Schoelz, W.C.HERAEUS GMBH&CO KG, MATERIALS TECHNOLOGY DIVISION, HERAEUSSTRASSE 12-14, 63450 HANAU, GERMANY

1. Introduction

To produce high purity Niobium (RRR ≥ 600) on an industrial scale, some points have to be considered. One of the factors that limits the RRR-values, in addition to the gas content, is the residual Ta content of the starting-material: the Ta content enriches during electron beam-melting and forms very stable carbides and nitrides, which makes the degassing process very difficult. Thus it is absolutely necessary to use a starting material with a low Ta content. Additionally it is possible to have a better degassing of the niobium by using appropriate changes during the melting process. On the other hand it is very importand to exclude all steps during processing, which return the gases or carbon to the material, e.g. welding etc. Also new getter-materials were tested during the last treatment like Ca. To control the very purity, it was necessary to create a special high precise referencematerial. This material was developed in cooperation with BAM Berlin and the working group for analytical chemistry of GdCh in Germany. Finally, it is also necessary to control the semifinished products like sheets or tubes before starting the expensive processing, to prevent failure of the finished cavities. That is the reason, why we have used Squids that are very sensitive and have a high resolution.

2. Correlation between RRR, the gas content and the Ta content

During the last year we made an extensive calculation based on 400 measured RRR samples and integrated them with a multiple regression over the Ta

Correlation between RRR, gas, and Ta content

content and the gas content. We discovered that RRR values >400 could only be reached by a Ta-content lower than 200 [µg/g]. The best method to produce such a raw material on an industrial scale is the liquid-liquid-extraction with MIBK or MIPK as showen in the following description:

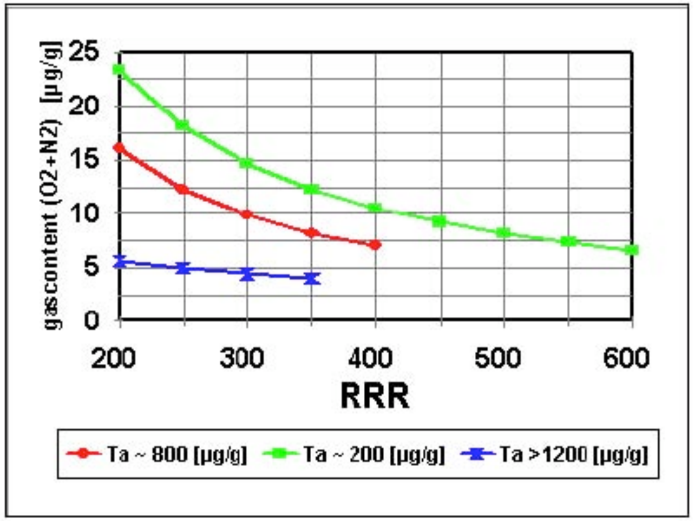
$NbCl_5 + TaCl_5 + MIBK / MIPK + H_2O$
Ta in the aqueous phase
Nb in the organic phase

$K_2NbOF_5 + K_2TaF_7 + MIBK / MIPK + H_2O$
Ta in the organic phase
Nb in the aqueous phase

After a cascade of 20 extractions the Ta content is down to about 200 [µg/g]. The theoretical value after 36 extractions is < 20 [µg/g].
So we produced more than 2 [to] of Nb with a Ta content of 200 [µg/g] in the standard sheet process and reached a RRR value better than 450 at once.

3. Degasing process during EB melting

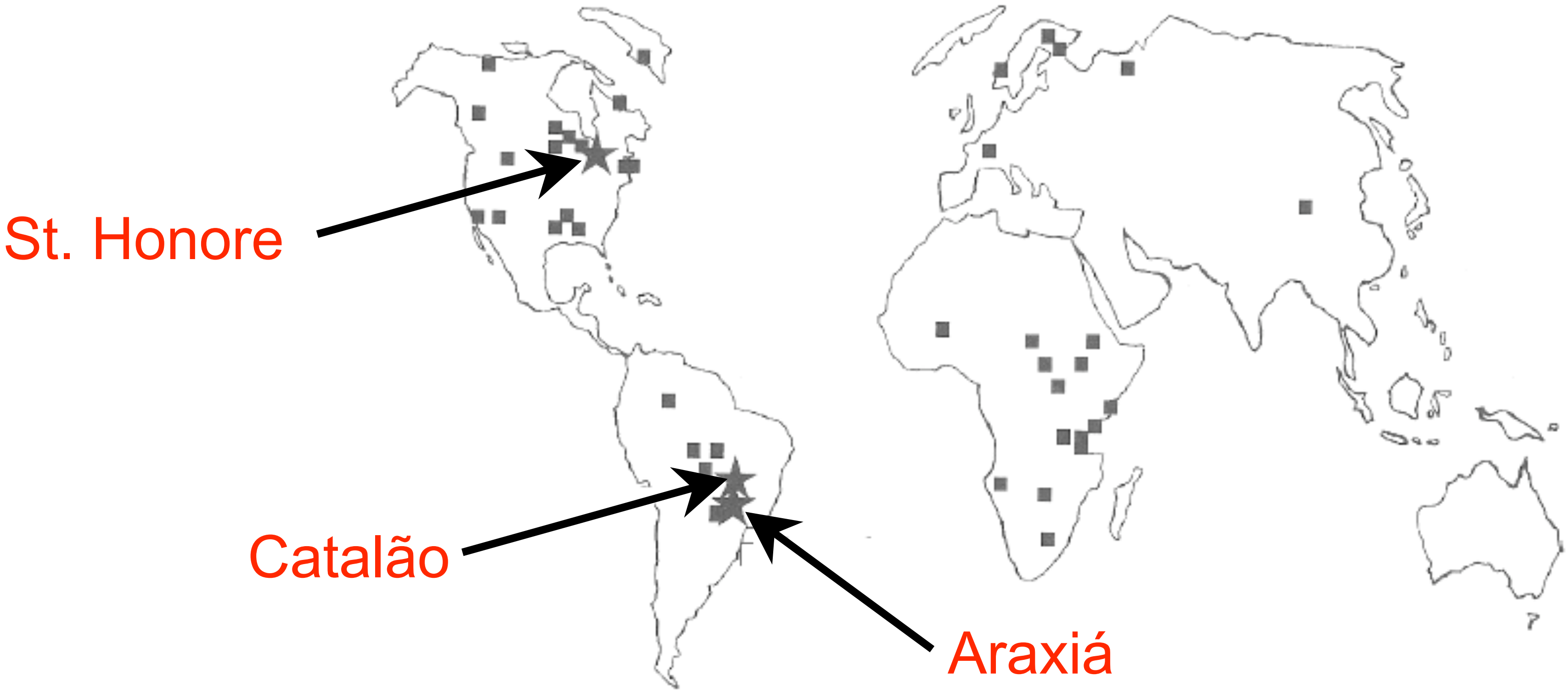
The purification and degasing process during electron beam melting is very complex. All metals with a lower melting point than Nb will be evaporated. Metals with a higher melting point like Ta and W will be enriched in the melt, because Nb also evaporates as Nb-oxides during the melting process. All gases are continuously in exchange with the hot metal surface. Gases from the furnace atmosphere will at first be adsorped on the metal surface and then dissolved in the metal or they produce evaporable products. Dissolved gases in the metal will diffuse to the surface and produce molecular



Correlation between RRR, gas, and Ta content

Nb mines

- Columbite $(Fe, Mn)(Nb,Ta)_2O_6$
- Tantalite $(Fe, Mn)(Nb,Ta)_2O_6$
- Pyrochlore $(Ca,Na)_2Nb_2O_6F$

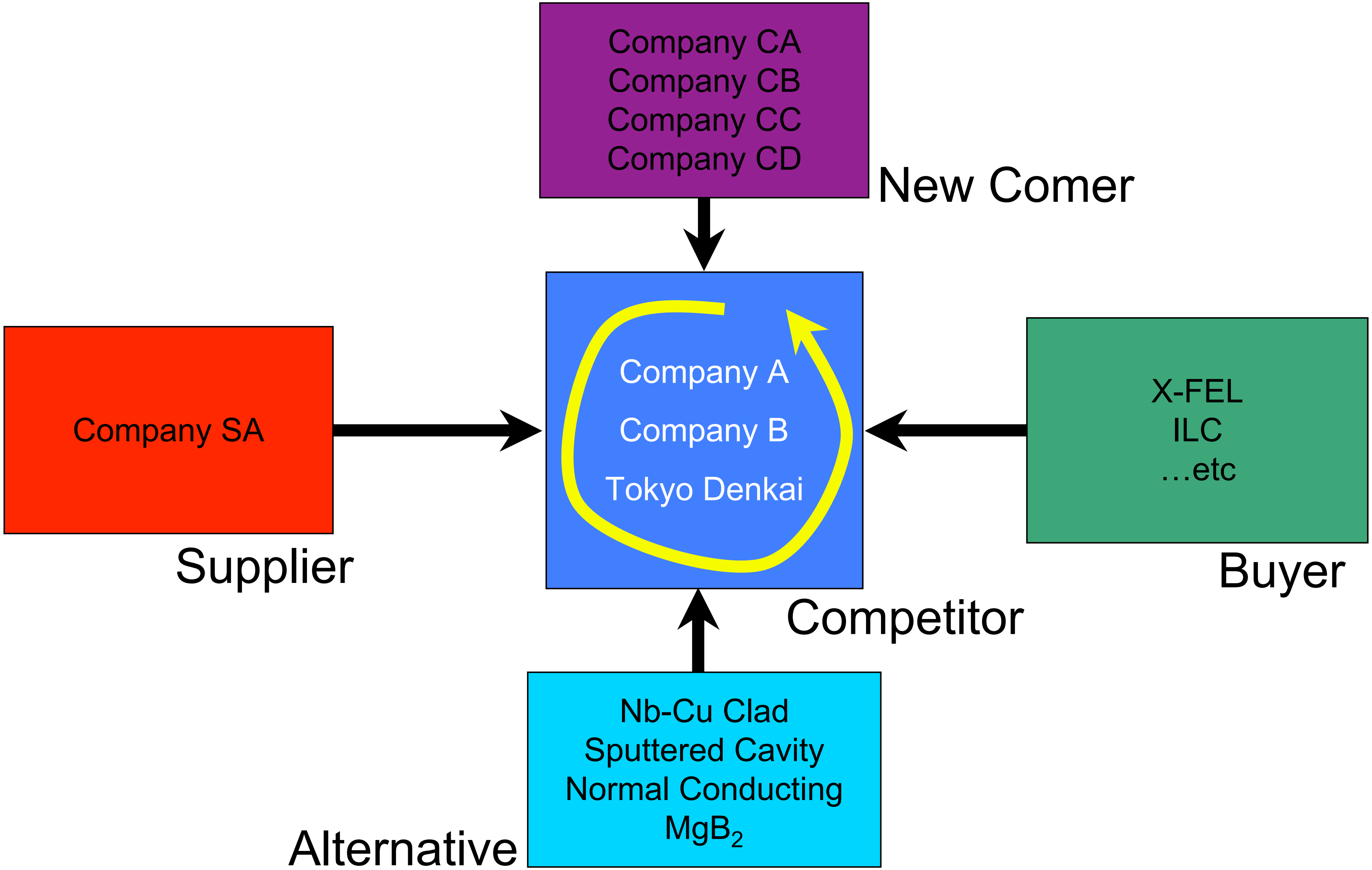


Imagumbai, et al: “Resource, production volume & manufacturing technology of niobium” Material Science & Technology Vol.72(2002)No.3 pp204-210

Bottleneck 2

Melting capacity

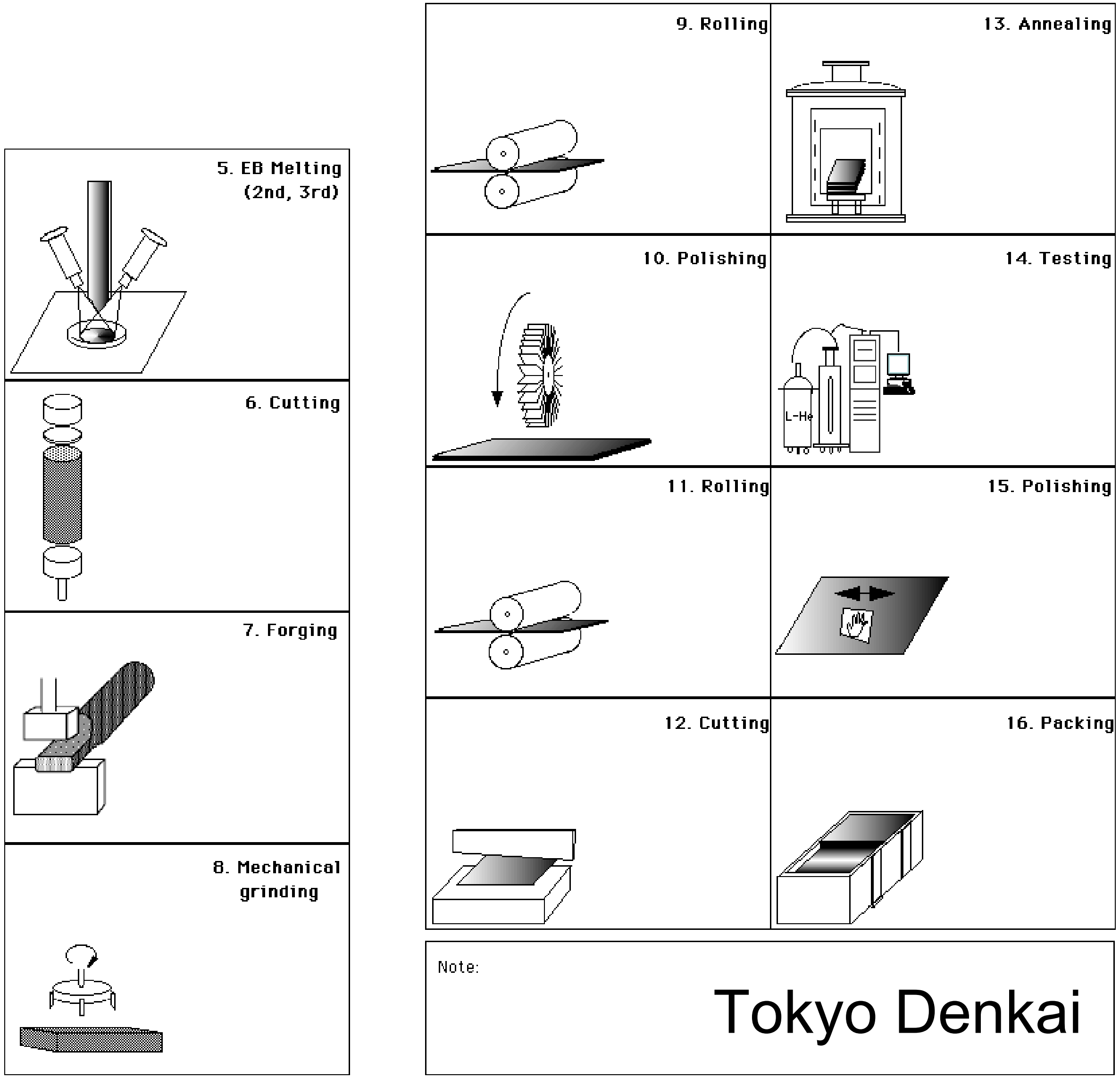
Five Force Analysis of Nb for SC Cavities



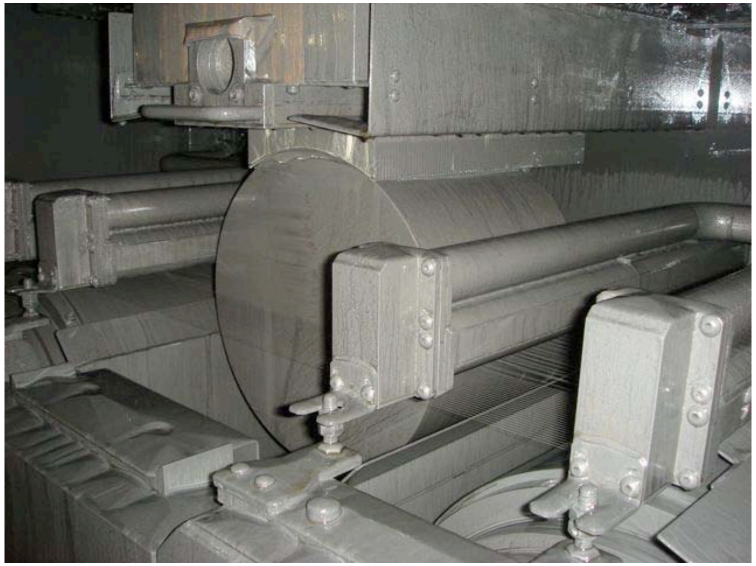
Bottleneck 3

Sheet production process

Sheet production process



Multi Wire Saw Slicing



Summary

- Small market
  - The demand for high purity Nb for superconducting accelerator is several tons per year, although the total Nb demand is 60 thousand tons per year.
  - Only three vendors are qualified for the XFEL project.
  - These three vendors have several main businesses. It can be said that it is difficult to concentrate the high purity Nb production even if only 3 years.
- All Nb vendors are buying Nb ingots from the same company
  - Producing high RRR Nb requires a raw material with a low Ta content. Today, only one company provides it.
- Sheet production
  - Sheet production is not easy. It needs experience. For example, material and shape of roll, timing of cross roll, intermediate annealing, grain size control, thickness irregularity…

Summary

- We have a time to consider to increase the high purity niobium production capacity.
- We would like to seek the solution.

Thank you for attention!