

# T I C

## TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

### PRESIDENT'S LETTER

Dear Friends,

As, for most of us, we move towards Springtime and the vagaries of the weather are matched by those of the World's regional economies, we can reflect upon the last few months since our General Assembly.

Prices of some precious metals used within the electronics industry have increased dramatically while others have fallen to new lows for this decade. We have seen new developments, such as Cabot Corporation proposing to take a stake in Sons of Gwalia, continuing the tantalum-niobium industry trend of further consolidation, integration and longer term strategic planning. Change continues to be the normal environment, and rather like the weather, something we have learnt to live with, however unpredictable.

As you all will know, we are planning to hold an Executive Committee meeting in Brussels on the morning of Tuesday April 21st. I would encourage you all to attend the luncheon to be held that day, and which is open to all members. This is your chance to put forward suggestions or ideas so that we can endeavour to make the T.I.C. of real and lasting value to its membership. Those who may not be able to join us, please send to the Secretary General soon your suggestions that you wish your Executive Committee to consider.

Planning for the Thirty-ninth General Assembly this year, in Prague, on October 11th to 13th is well advanced and will be reported upon at our meeting in April.

Sincerely,

Bill Millman

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### HYDROMETALLURGICAL EXTRACTION OF TANTALUM AND NIOBIUM IN CHINA

*This paper by Mr He Jilin, Mr Zhang Zongguo, Mr Xu Zhongting, Mr Nie Dajun and Mr Li Bin, of Ningxia Non-ferrous Metals Smelter, was presented at the T.I.C. meeting in Xian, China, in October 1997.*

#### GENERAL

In China, research work on hydrometallurgical extraction of tantalum and niobium began in 1958 at Beijing General Research Institute for Non-ferrous Metals. The technology consisted of caustic fusion of the ore and fractional crystallization of complex fluorides of tantalum and niobium. Later on, some research work was conducted on chlorination for preparation of Ta/Nb compounds. In 1962, work began on Ta/Nb separation by solvent extraction. In 1964, a technology of HF-H<sub>2</sub>SO<sub>4</sub>-MIBK (methyl isobutyl ketone) process was introduced into production. A factory, namely Ningxia Non-ferrous Metals Smelter (NNMS) was then founded in 1965 in Ningxia Autonomous region, with an annual capacity of 15 tons of potassium fluotantalate and 4 tons of niobium pentoxide.

In the past three decades we have continued to improve our hydrometallurgical processing of tantalum/niobium extraction, and significant progress has been made. Technology improved in many aspects, and production capacity increased to a large scale. Currently there are five major plants in China that are engaged in hydrometallurgical processing of tantalum and niobium concentrates. A brief description of these plants is given in Table 1.

Plant	Address	Capacity (ton/year)				
		K <sub>2</sub> TaF <sub>7</sub>	Nb <sub>2</sub> O <sub>5</sub> Industrial	Ta <sub>2</sub> O <sub>5</sub> Industrial	Nb <sub>2</sub> O <sub>5</sub> High Purity	Ta <sub>2</sub> O <sub>5</sub> High Purity
Ningxia Nonferrous Metals Smelter	Shizuishan City Ningxia Autonomous Region	180	60		5	3
Jiujiang Nonferrous Metals Smelter	Jiujiang City, Jiangxi province	100	40	20	20	10
Ta-Nb Division of Zhuzhou Hard Metal Factory	Zhuzhou City, Hunan Province	100	40	30		
Tsonghua Smelter	Guangzhou City Guangdong Province	80	80			
Ta-Nb Hydro-metallurgical Plant of Limu Tin Mine	Limu, Guangxi Autonomous Region	60	30			
<b>Total</b>		<b>520</b>	<b>250</b>	<b>50</b>	<b>25</b>	<b>13</b>

Table 1: Five major plants that are engaged in hydrometallurgical extraction of tantalum and niobium

All these five plants use a similar process in extracting and separating tantalum and niobium as shown in Figure 1. The concentrates, finely ground in a vibrating ball mill, are digested with HF-H<sub>2</sub>SO<sub>4</sub> acids. The resulting solution, after conditioning for acidity, is treated with organic solvents (MIBK or 2-octanol). Tantalum and niobium are extracted into the organic phase, while most of the impurities such as Fe, Si, W, Ti, and Mn remain in the aqueous slurry which is then discarded. The organic solution is then washed with acid solution to be purified further. A niobium-stripping solution is used to extract niobium from the acid washed pregnant solution into a solution of oxyfluoniobic acid. Then a tantalum-stripping solution is applied to extract tantalum from the niobium-stripped organic phase solution, resulting in a solution of fluotantallic acid. To produce potassium fluotantalate, the fluotantallic acid solution is heated, conditioned in acidity, and a potassium salt (KCl or KF) is added to it. Crystallized potassium fluotantalate is used as starting material for sodium reduction to produce tantalum powders.

The solutions of fluotantallic acid and oxyfluoniobic acid are also used to make pentoxides of tantalum and niobium respectively by ammonia precipitation, followed by filtration; the precipitates are dried and calcined.

The pentoxides are used to produce metals by thermo-reduction with carbon or aluminum. Also, the oxides are important raw materials in production of ceramic capacitors, optical glasses and single crystals of lithium tantalate or niobate.

## FEATURES OF THE TECHNOLOGY AND EQUIPMENT

China's tantalum and niobium processors pay much attention to technical progress. They have long been striving to improve their technology and equipment, which have now reached a fairly good condition. It is not difficult to see the advances made by the hydrometallurgists here in China by reviewing the features we describe below.

### 1. Adoption of HF-H<sub>2</sub>SO<sub>4</sub> system

All the processors in China have adopted a mixed HF-H<sub>2</sub>SO<sub>4</sub> acid system to open the ore. H<sub>2</sub>SO<sub>4</sub> in the system lowers the partial pressure of HF, reduces its volatilization loss, thus decreases its consumption and hence helps improve environmental conditions. H<sub>2</sub>SO<sub>4</sub> also enhances the digestion of the ore, making some minerals easier to dissolve than in a single HF solution, thus the over-all recovery of tantalum/niobium may increase. It is true that a single HF system might have some advantages over the mixed HF-H<sub>2</sub>SO<sub>4</sub> system, but the practice of our Chinese processors shows that the introduction of H<sub>2</sub>SO<sub>4</sub> into the acid system has more merits than deficiencies.

### 2. Use of MIBK or 2-octanol as extractant

Ningxia Smelter uses MIBK as extractant, while the processors in southern China choose 2-octanol.

The comparison of physical properties and hydrometallurgical performance of the two extractants is shown in Tables 2, 3, 4, and 5.

	Formula	Molecular weight	Density g/cm <sup>3</sup>	Boiling point °C	Scintillation point °C	Viscosity cp	Solubility in water %
MIBK	C <sub>6</sub> H <sub>12</sub> O	86.0	0.7978	116	23	1.803X10 <sup>-3</sup> (30°C)	1.8 (by volume)
2-octanol	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>5</sub> CHOHCH <sub>3</sub>	130.23	0.82	175	73	1.34X10 <sup>-2</sup> (20°C)	0.08 (by weight)

Table 2: Physical properties of MIBK and 2-octanol

	H <sub>2</sub> SO <sub>4</sub> , N					
	0.5	1.0	1.5	2.0	3.0	5.0
MIBK	6374.5	5940.2	3074.8	3074		
2-octanol	148.0	146.5	152.8	177.3	212.0	80.5

Table 3: Separating ratio of Ta to Nb by MIBK and 2-octanol, in HF-H<sub>2</sub>SO<sub>4</sub> system

Notes: Pregnant solution for MIBK extraction: (Ta,Nb)<sub>2</sub>O<sub>5</sub> content = 100 g/l; Ta:Nb = 1.05; HF concentration = 2N  
Pregnant solution for 2-octanol extraction: (Ta,Nb)<sub>2</sub>O<sub>5</sub> content = 100 g/l; Ta:Nb = 1; HF concentration = 1N

	H <sub>2</sub> SO <sub>4</sub> , N							
	2	4	5	6	7	8	9	10
MIBK			1.5	1.8	3.5	6.5	10.3	20.0
2-octanol	1.02	1.96		4.85		17.2		40.2

Table 4: Comparison of extraction ratio of tungsten by MIBK and 2-octanol

Notes: Pregnant solution for MIBK extraction: (Ta,Nb)<sub>2</sub>O<sub>5</sub> content = 100 g/l; Ta:Nb = 1.05; W = 0.55 g/l; HF concentration = 6N  
Pregnant solution for 2-octanol extraction: (Ta,Nb)<sub>2</sub>O<sub>5</sub> content = 96.23 g/l; Ta:Nb = 2; W = 4.1 g/l; HF concentration = 6.5N

	Saturation volume g/l	HF, N	H <sub>2</sub> SO <sub>4</sub> , N
MIBK	323.6	4.64	1.49
2-octanol	226.0	3.05	1.14

Table 5: Comparison of saturation volume (combined Ta<sub>2</sub>O<sub>5</sub>+Nb<sub>2</sub>O<sub>5</sub> content) of MIBK and 2-octanol, in HF-H<sub>2</sub>SO<sub>4</sub> system

Notes: Composition of original solution (g/l): Ta<sub>2</sub>O<sub>5</sub> = 82.6, Nb<sub>2</sub>O<sub>5</sub> = 81.0, Fe<sub>2</sub>O<sub>3</sub> = 13.27, WO<sub>3</sub> = 2.52, SiO<sub>2</sub> = 0.45, TiO<sub>2</sub> = 1.71, HF = 4.56N, H<sub>2</sub>SO<sub>4</sub> = 7.45N

Although these two extractants differ in their physical and extractive properties, they both give satisfactory results when the process parameters are carefully controlled. Both of them can be used in production of industrial grade potassium fluotantalate and niobium pentoxide. They are both capable of being used for production of high purity niobium pentoxide and tantalum pentoxide.

With its low volatility at ambient temperature, low solubility in water and low price, and a well supplied market, 2-octanol attracted great attention when it first succeeded in experimental use at the Ningxia Smelter in 1975.

Currently it is used at all plants in southern China, but Ningxia Smelter itself chooses MIBK for its unique physical properties and extraction performance, and also for its universal compatibility with environmental conditions.

### 3. Use of slurry extraction

In 1970, Ningxia Smelter succeeded in developing the technology of continuous slurry extraction, featuring the direct extraction of valuable metals from slurry, which is a mixture of solid and liquid resulting from ore digestion. Such a process saves the operator the very troublesome filtration step, raises production efficiency, and increases equipment capacity. It offers great ease in conjoining the pregnant organic solution flow with the downstream operation, and makes the process a continuous one. At the same time, a higher degree of enrichment could be realized. And its advantage is even more manifest in treating low grade ores. This technique has been adopted by all the processors in China's tantalum and niobium industry.

### 4. Cold crystallization of potassium fluotantalate

The traditional technique involves heating fluotantalate acid solution up to 85°C, adding HF and KCl, then cooling it down to form a supersaturated solution from which crystals of  $K_2TaF_7$  are produced. In 1991, Ningxia Smelter succeeded in experiments on cold crystallization of potassium fluotantalate. This technique features a short cycle, high capacity and continuous operation. The low crystallization temperature and low acidity involved make it easier to deal with corrosion problems in the equipment and thus to reduce the contamination of the products from equipment materials. Lower free HF content in  $K_2TaF_7$  products eases corrosion problems during sodium reduction, which will lower iron, nickel and chromium content in tantalum powders. Furthermore, the yield is higher, energy consumption is lower and the operating conditions could also be improved. All these factors make it worthy of practical application. In 1993, a pilot production line of continuous cold crystallization was built, with a capacity of 30 tons of potassium fluotantalate per year.

### 5. Oxide production

In 1993, Ningxia Smelter succeeded in developing a new method of continuous spray precipitation (Figure 2) to produce low fluorine tantalum and niobium oxide. This method gives larger grains of precipitate, with low content of fluorine, ease of washing and little water remaining in the filtered cake. This method gives oxides of high bulk density and good flow ability after calcination. And it is a continuous process. It needs only a small tank, with raw materials reacted and precipitated in a short time.

In order to manufacture high purity oxide products with low fluoride content, Ningxia Smelter developed a hot hydrolysis technique. In this process, a continuous flow of live steam passes through the hydroxide, placed in the crucibles in the furnace (Figure 3), and heated up to 700~800°C. Under such conditions, the fluoride remaining in the raw material will be converted into gaseous HF and driven off from the oxide material, thus the fluoride content in the product oxide is lowered to less than 10ppm.

In 1983, Zhuzhou Hard Metal Factory successfully developed a new grade of niobium oxide, for use in high frequency ceramic capacitors. It well met the demands of the domestic market and some of the product was exported abroad.

Beijing General Research Institute for Non-ferrous Metals developed an oxalate crystallization technique in 1985 to produce high purity niobium oxide. This kind of niobium oxide was a so called 'no fluorine product' which made a further step towards meeting the demand of the high-tech field. The flow sheet of this process is shown in Figure 4.

## 6. Extraction equipment

In 1970, Ningxia Smelter developed a new design for extractors for continuous slurry extraction. It is small in size, but large in capacity, as it can treat 80 kg of Ta/Nb oxides per hour.

In 1991 the extraction/separation equipment was further improved, and a new model of high flow mixer/settler box was developed. The capacity was six times greater than the original design but the geometric dimensions were kept unchanged.

Based on years of experience of box extractor application, and taking advantage of a tower extractor, Ningxia Smelter developed a combinatory extractor in 1993. Its stage efficiency increased to more than 95%. It basically eliminated the third phase in the settler, and effectively controlled the interface and liquid levels.

Efforts were made to stabilize extraction technology at Jiujiang and Conghua Smelters and Zhuzhou Hard Metal Factory. Various steps were taken to improve extractor design and stirring mode as well. They developed various flow-controlling devices which stabilized their technology and increased their output.

## 7. Equipment for drying and calcination

Ningxia Smelter imported and used a vacuum rotary dryer for drying K salt to replace the outdated steam heated wooden installations, in 1992. It reduced volatile impurities in potassium fluotantalate by 75% and increased output and upgraded product quality.

Zhuzhou Hard Metal Factory has developed a continuous far-infra-red dryer, which has increased the productivity and turned the process into a continuous one. Ningxia Smelter, Jiujiang Smelter and Zhuzhou Hard Metal Factory have all used rotary kilns in place of resistant muffles for calcination of Ta-Nb oxides. It eased labour intensity and raised both production output and product quality.

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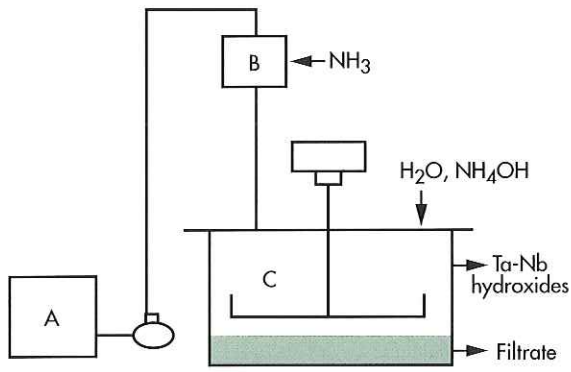
## QUALITY OF PRODUCTS

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Some 20 years ago there were only two hydrometallurgical products - potassium fluotantalate and niobium pentoxide - in China's tantalum and niobium market. Now the product list has expanded to include three series and eight grades, i.e. potassium fluotantalate, tantalum pentoxide (industrial, optical glass and high purity grades) and niobium pentoxide (industrial, optical glass, high purity and ceramic grades). As the processors insist on continually advancing their technique and equipment, the quality of their products has been improving year by year. Taking Ningxia's  $K_2TaF_7$ , for example, the total content of ten common impurities (Fe, Ni, Cr, Nb, Si, Cu, W, Mo, Mg, Ti) in it in 1996 was reduced to about a quarter of that in 1981 (see Figure 5).

The industrial grade niobium oxide has gained similar upgrading (see Figure 6). And the high purity niobium oxide has also made great progress (see Figure 7).





A - Tank for Ta-Nb solutions  
 B - Spray-precipitator  
 C - Vessel for filtration and washing

Figure 2: Sketch of spray-precipitation

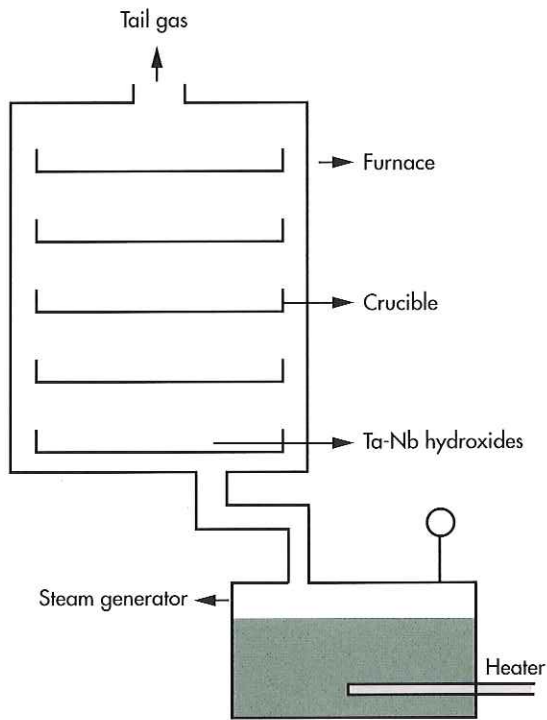


Figure 3: Defluorination by hot hydrolysis

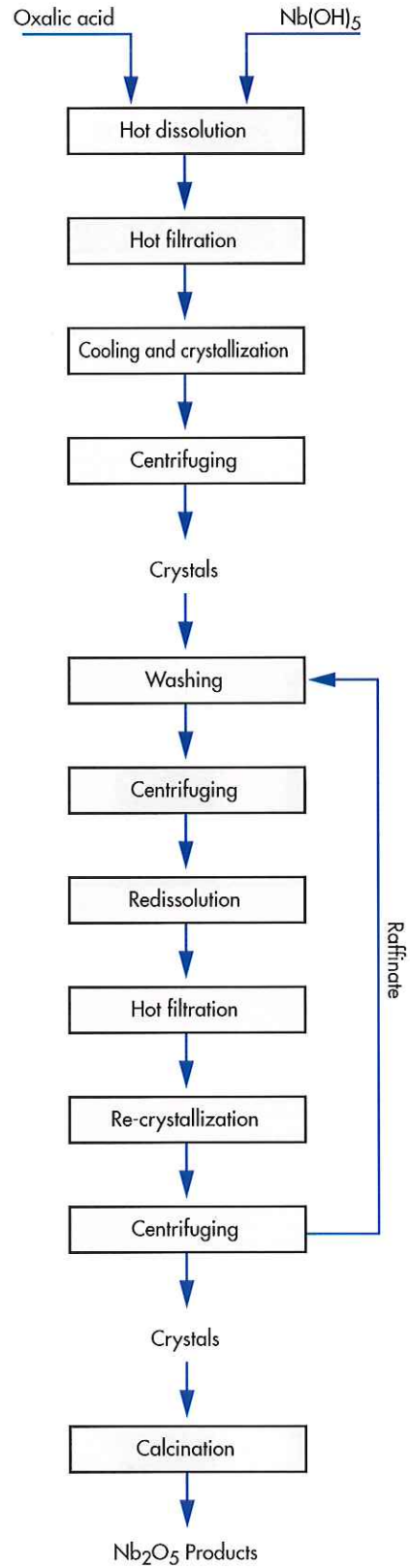


Figure 4: Oxalate crystallization process

## THE NEGOTIATIONS, START-UP AND OPERATION OF A HIGH TECHNOLOGY MANUFACTURING JOINT VENTURE IN CHINA: A PERSONAL PERSPECTIVE

Mr Arthur J. Yarzumbeck, General Manager, Shenzhen GKI Electronics Co., Ltd, an IBM China and Great Wall Computer Group joint venture, presented this paper at the T.I.C. meeting in Xian in October 1997.

### ABSTRACT

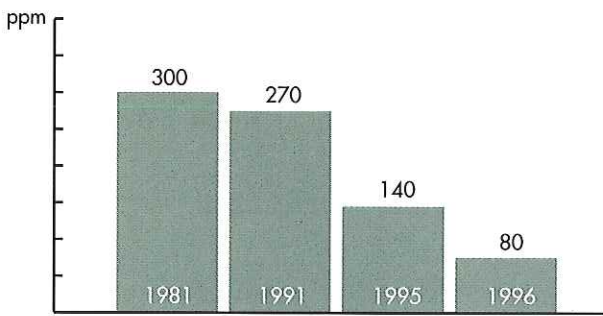
Many multi-national corporations have recognized the enormous potential of conducting operations in China. Balancing the benefits are the well (and sometimes not so well) publicized pit-falls of doing business in an economy emerging from approximately 50 years of fundamentalist communist rule as well as displaying cultural traditions enigmatic to typical western management practices. This short presentation will highlight the personal experiences of an American expatriate general manager from a large multi-national corporation, IBM, in the negotiations, initiation and continuing operations of a technology based manufacturing joint venture for electronic cards, successfully conducting business from a factory in the Shenzhen SEZ.

### GKI MISSION

Shenzhen GKI Technology Co., Ltd. is a technology joint venture located in the People's Republic of China, which provides high quality, competitively priced manufacturing services to our customers worldwide.

### Chronology

1994:	June	Began partner selection
	October	Equipment set/process defined (Starfire family)
	December	Completed contract negotiations
1995:	April	Contract signed
	May	Business license obtained
	July	Line qualified
	August	First article manufactured
1996:	November	Completed SSD Starfire MRR
	1/2Q	Qualified/ramp up Token ring Keyboard card and ESS (PCCO)
	June	First profitable month
	August	Break-even for the year
1997:	December	Began Scorpion qualification
	1Q	Continued Scorpion qualification volume production
		Completed Tango qualification/ramp up
		Completed Intel qualification/ramp up
		Obtained ISO-9002 Certification
		Capacity expansion
2Q		Began Tango production
3Q-4Q		Capacity and capability expansion



5: Total content of ten common impurities in  $K_2TaF_7$ , in ppm

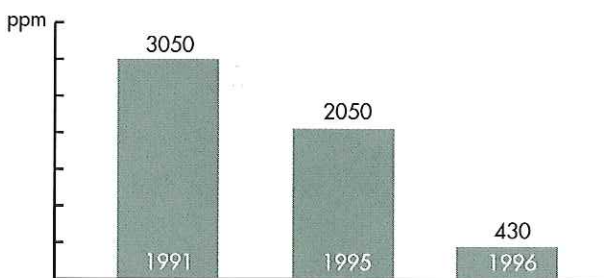


Figure 6: Impurity (Ti, Si, Fe, W) content of industrial grade  $Nb_2O_5$ , in ppm

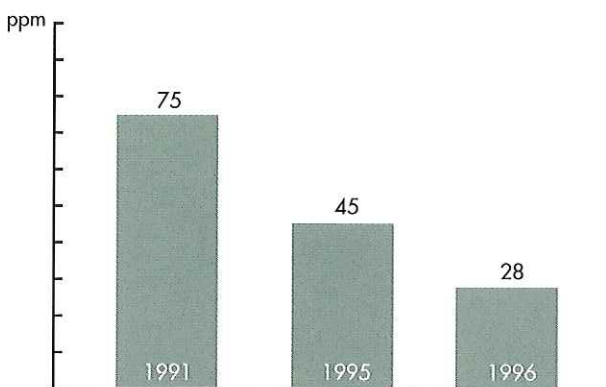


Figure 7: Impurity (Ta, Si, Fe) content of high purity  $Nb_2O_5$ , in ppm

#### Overview (1)

- Sponsoring division: IBM Microelectronics
- Type of enterprise: equity joint venture
- Partners: Great Wall Computer Group and IBM
- Registered capital: \$US 10M
- Date of business license: 9th May 1995
- Export sales license: allowed 85% on-shore
- Technology transfer agreement: access to IBM ECAT (electronic cards) technology with on-site assistance from lab or plant of control

#### Overview (2)

- Status:
- 670 employees (85% direct)
- 2.5 expats
- 1.1M card assemblies (96) → 2.4M (97)
- Will double capacity within current facility

#### Certifications:

- ISO-9002 (February 97)
- Advanced Technology Co. (SEDB)
- Advanced & New Technology Co. (SSB)
- 1997 Customs Credible Company

#### Sister sites:

- IBM Yasu ECAT (Japan)
- IBM Charlotte ECAT (USA)
- IBM Vimercate ECAT (Italy)
- IBM APD Laboratory (USA)

#### Major customers:

- IBM Mid Range File Facility, Singapore
- IBM Personal Computer Division, Japan
- IBM Networking Systems Division, USA
- IBM Low End File Facility, Hungary
- Intel (Great Wall licensee for M/B)
- IIPC
- Great Wall

When contemplating a joint venture in China, always remember an old Chinese proverb: 'A journey of 1,000 Li begins with one step'.

## GWALIA CONSOLIDATED RESTRUCTURES

*Gwalia Consolidated Ltd ('GWC') and Sons of Gwalia Ltd ('SGW') recently announced a corporate re-structuring and consolidation of core assets.*

Key points of the transaction are as follows:

- GWC, pursuant to a Scheme of Arrangement, to distribute all of its approx. 17.9 million shares in SGW plus a capital return of \$6 million to its shareholders (other than SGW) funded from GWC's share premium reserve.
- GWC shareholders (other than SGW) will receive two SGW shares plus seventy cents (70c) in cash in exchange for the cancellation of every seven GWC shares held by them
- GWC will become a wholly controlled entity of SGW and will subsequently be delisted from the Australian Stock Exchange
- SGW will not need to issue any new shares as part of the corporate restructure with GWC but will consolidate GWC's assets and liabilities into its balance sheet following the restructure.

Tantalite contracts now total A\$350 million and A\$30 million placement to Cabot Corporation.

SGW and GWC also announced two important and related corporate initiatives:

- GWC has extended its existing contracts with its two major customers, at increased prices, for the sale of all of its budgeted tantalite production, from its Greenbushes and Wodgina Mines, from the year 2000 to 2003 and has granted options for further extensions. The contracts for the years 1998 to 2003 (inclusive) now have a gross value of A\$350 million. GWC will continue expansion and development of these mines to ensure a continued supply of tantalite under these contracts.
- Subject to completion of the corporate restructure, Cabot Corporation, a United States corporation which is one of GWC's largest tantalite customers, will acquire an approximate seven per cent (7%) interest in SGW by taking a placement of 7.5 million ordinary shares at \$4 per share raising \$30 million in cash. Cabot Corporation is a large North American chemical and strategic metals processor. It is the world's largest producer of Carbon Black and is one of the two largest tantalum processors. It also has substantial interests in natural gas distribution in New England and in super-alloy production. Cabot is listed on the New York Stock Exchange and has a market capitalisation of approximately US\$1.9 billion.

### SGW FOLLOWING RE-STRUCTURING

Upon completion of the re-structuring and consolidation, SGW's Gold and Industrial Minerals Divisions would comprise the following:

#### Gold

- Sons of Gwalia Mine
- Marvel Loch Mine
- Yilgarn Star Mine (70%)
- Bullfinch Mine
- Laverton Mine

#### Industrial Minerals

- Greenbushes Tantalum Mine
- Wodgina Tantalum Mine

- Greenbushes Lithium Mine
- Kemerton Silica Sand Mine (70%)
- Mt Seabrook Talc Mine (50%)

Minor assets will be sold.

The Executive Chairman of Sons of Gwalia Mr Peter Lalor stated: 'I believe substantial benefits will flow to shareholders of both companies including increased earnings and cash flows from the merged assets, increased management focus in managing one company only and the direct access to the cash flows and earnings from both the gold and industrial minerals projects as against the current passive investments held by both companies in each other'.

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## LETTER

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Following the meeting in Xian, the T.I.C. received this kind letter from Mr Yarzumbeck of Shenzhen GKI Electronics:

I wanted to take a few moments to express my thanks for the hospitality you extended to my wife and myself at the Thirty-eighth T.I.C. General Assembly in Xi'an, PRC, from 6 to 8 October 1997.

I truly enjoyed the opportunity to discuss my experiences in establishing and running a technology joint venture in China as your guest speaker. However, the real enjoyment was the chance to meet and interact with the various members during the meetings, at dinner and during the wonderful social engagements and tours that had been arranged. I would especially like to thank Judy and George for the great co-ordination that went into making this a very special event for my wife and myself.

I would like to extend an invitation to you to visit GKI when you are in this part of the world. It would be my distinct pleasure to host you in my organization and have you meet my great Chinese team.

Arthur J. Yarzumbeck, General Manager

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## MEMBER COMPANY NEWS

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### NEC

Mr Tetsuo Suzuki, Senior Manager of the Engineering Department of Energy Devices Division, has taken over from Mr Yoshihiko Saiki as the company's delegate to the T.I.C.

### Siemens Matsushita

The Heidenheim plant of Siemens Matsushita has been expanded, and production capacity for tantalum electrolytic capacitors in cases C, D and E has been increased by 200 million components per year. The company has also issued a new short form catalogue of 'Passive components for surface mounting': the 1998 edition is available from Siemens' offices or can be viewed on the Internet under <http://www.siemens.de/pr/index/htm>.

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Tel.: (02) 649.51.58  
Fax: (02) 649.64.47  
Please note that there is no longer a telex line  
to 40 rue Washington

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