## TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

#### PRESIDENT'S LETTER

Friends,

You will receive this Bulletin just prior to the General Assembly meeting in Greenville, South Carolina. Over the past three months, the demand for tantalum capacitors has continued to be soft. The slowing growth in personal computers and significant overbuilding that occurred in 1995 and early 1996 has not completely worked its way through the system. Hopefully, this correction will be short lived.

On a more personal note, I have resigned from Cabot effective the first of August and have likewise resigned as the President of the T.I.C. Dave Maguire of Kemet has kindly agreed to act as interim President and chair the Executive Committee meeting and the Thirty-seventh General Assembly in South Carolina. He hopes to see you all at this meeting.

I wish you all well for the future, and encourage you to stay in touch.

Sincerely,

R.S. Barron

On behalf of the Executive Committee and all the member companies of T.I.C., I would like to express our appreciation to Bob Barron for his services to the Executive Committee of T.I.C. and as President since October 1995. Bob Barron took a very active role in T.I.C.'s affairs and was very helpful in the preparations for the Thirty-seventh General Assembly and associated meetings. All of us wish him well.

D.E. Maguire Acting President

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### GREENVILLE, OCTOBER 20TH-22ND 1996

This year's General Assembly meeting will be hosted by Kemet Electronics in Greenville, South Carolina, U.S.A. A welcome reception will open the meeting on the evening of Sunday October 20th. Monday will be devoted to the Thirty-seventh General Assembly and an appealing programme of technical presentations, with a gala dinner to which all delegates will be invited as the guests of Kemet. On the morning of October 22nd there will be a plant tour of Kemet's capacitor facility, and the meeting will close with lunch on that day.

Invitations have been sent to member company delegates, anyone else interested in attending should contact as soon as possible the Secretary General of the T.I.C., 40 rue Washington, 1050 Brussels, Belgium, as the deadline for pre-registrations and hotel bookings is on September 24th.

#### Technical programme:

Developments in tantalum and niobium during the last year by Dr George Korinek, Technical Adviser to the T.I.C.

Superconductor developments and applications in Japan, based on niobium

by Professor K. Tachikawa, Tokai University, Kanagawa, Japan

The use of niobium in low temperature superconductors - short and long term market projection

by Mr Dave Alderson, IGC Advanced Superconductors, Waterbury, Connecticut, U.S.A.

Experience in the manufacture of Nb-Ti alloys for superconductors

by Mr Tadeu Carnero, Reference Metals Company Inc., Pittsburgh, Pennsylvania, U.S.A.

The future of tantalum ore supply by Mr Richard O. Burt, Tantalum Mining Corporation of Canada Ltd., Lac du Bonnet, Manitoba, Canada

New design of tantalum capacitors, which allows small size down to 0603, with high volume efficiency by Mr Ian Salisbury, AVX Ltd, Paignton, Devon, England

A comparison of applications for tantalum, aluminum and multilayer ceramic capacitors

by Mr John Primax, Kemet Electronics Corporation, Greenville, South Carolina, U.S.A. The use of tantalum capacitors in the PC industry by Mr Bill Schaefer, IBM Corporation, Raleigh, North Carolina, U.S.A.

Tantalum capacitor trends in the hard disk drive industry by Mr Reginald L. Hofmaier, Seagate Technology, Oklahoma City, Oklahoma, U.S.A.

Expectations and cost drivers for capacitors in the automotive industry

by Mr Steven Bacha, Ford Motor Company, Detroit, Michigan, U.S.A.

#### TANTALUM INDUSTRY IN CHINA

by Mr Ma Fukang and Professor Chu Youyi, of the Nonferrous Metals Society of China, and Mr Zeng Fangping, of Ningxia Nonferrous Metals Smeltery.

This paper was presented to the 125th Annual Meeting of TMS in Anaheim, California, February 4th-8th 1996, and will be published in the Proceedings of the Tantalum Symposium which took place during that meeting. We should like to thank TMS, The Minerals, Metals & Materials Society, for permission to re-print it here.

#### **ABSTRACT**

The tantalum reserves in the mines of China are estimated to be 39 840 tonnes Ta<sub>2</sub>O<sub>5</sub>, accounting for about 12% of the total world reserves. The tantalum industry in China was founded in the 1960's. Various tantalum compound and metal products are produced in five major plants. The application of tantalum in capacitors dominates domestic tantalum consumption. Great efforts have been made to improve the production techniques of tantalum powder and wire for capacitors. Recently, tantalum powder with a CV value reaching 50 000 µFV/g and tantalum wire with diameter 0.2 mm or less can be supplied to domestic and international markets. However, up to now the total sales amount of tantalum capacitors produced in China can only meet about 30% of domestic demand. China's tantalum industry foresees a prosperous future.

#### INTRODUCTION

The tantalum industry in China was founded in the 1960's. In 1964 Ningxia Nonferrous Metals Smeltery was first set up. Up to now, various kinds of tantalum compound and metal products have been produced which can basically meet the domestic demands. The annual production capacities of potassium fluotantalate and tantalum powder have reached more than 500 and 100 tonnes, respectively. In 1992, capacitor grade tantalum powder of Ningxia Nonferrous Metals Smeltery entered the international market, demonstrating that Chinese tantalum products have been improved to a new level of quality.

#### **RAW MATERIALS**

As shown in Table I, tantalum mines in China are widely distributed in Jiangxi, Xingjiang, Guangxi, Guangdong, Hunan and Fujian Provinces. The total tantalum reserves are estimated to be 39 840 tonnes Ta<sub>2</sub>O<sub>5</sub> and account for about 12% of the world total reserves and rank China after CIS and Australia, thus in the third position in the world. But the grade of the deposits is low, mostly containing 0.016% Ta<sub>2</sub>O<sub>5</sub> or less, thus beneficiation is important to upgrade the concentrate. Continuous efforts have been made to improve the recovery ratio and grade of tantalum concentrate.

Province	Mine	Reserves	
Jiangxi	Yichun	17 650	
· ·	Shicheng	350	
	Dajishan	2 500	
Xingjiang	Keketuohai	170	
0. 0	Aletai	300	
Guangxi	Limu	2 300	
Guangdong	Hengshan	700	
	Boluo	5 420	
Hunan	Xianghualing	5 530	
	Xiangdong	3 100	
Fujian	Nanping	1 820	
Total		39 840	

Table 1: Tantalum reserves in the mines of China (tonnes Ta<sub>2</sub>O<sub>5</sub>)

Nowadays most of the tantalite supplied as raw materials for tantalum producers in China comes from Yichun Mine in Jiangxi, and Keketuohai Mine and Aletai Mine in Xingjiang. In some cases tantalum-containing tin slags and imported concentrates and struverite are used.

#### **TANTALUM PRODUCERS AND PRODUCTS**

There are five main tantalum producers in China. They are:

- Ningxia Nonferrous Metals Smeltery (NNMS) in Ning-Xia Autonomous Region
- Jiujiang Nonferrous Metals Smeltery (JNMS) in Jiangxi Province
- Zhuzhou Cemented Carbide Works (ZCCW) in Hunan Province
- Conghua Smeltery (CHS) in Guangdong Province
- Limu Tin Mine (LTM) in Guang Zhuang Autonomous Region.

The main tantalum products of these plants are listed in Table II. Among them Ningxia Nonferrous Metals Smeltery is the most important production base and research centre for tantalum in China. The production capacities of NNMS for K<sub>2</sub>TaF<sub>7</sub>, tantalum powder and wire account for 60-70% of the national total.

The production flowsheet for tantalum main products is shown schematically in Figure 1. All the five producers run their processes beginning with ore digestion in the H<sub>2</sub>SO<sub>4</sub>-HF system. But the extractants used for extracting the valuable metals and separating tantalum and niobium from each other are different: NNMS uses MIBK (methyl isobutyl ketone), while the others use 2-octanol.

Plant	$K_2$ TaF $_7$	Ta <sub>2</sub> O <sub>5</sub>	Powder	TaC	EB ingot	Wire	Others*
NNMS	+	+	+	+ ,	+	+	+
JNMS	+	+	+	+		+	+
ZCCW	+	+	+	+	+	+	+
CHS	+	+	+	+			
LTM	:+:	+	+	+			

<sup>\* &#</sup>x27;Others' include plates, strips, foils, rods, tubes, etc.

Table II: Tantalum producers and products in China

Potassium fluotantalate (K<sub>2</sub>TaF<sub>7</sub>) is crystallised out with KCl, and most of this K-salt is used in home plants as an intermediate product to produce tantalum powder by the sodium reduction method. But some producers sporadically export K<sub>2</sub>TaF<sub>7</sub> to the international market, and in recent years there seems to have been an increasing trend towards this exportation. The improvement of production technology and new products both for K<sub>2</sub>TaF<sub>7</sub> and tantalum powder will be mentioned in detail in the next part of this paper.

The majority of tantalum powders are produced for tantalum capacitor applications, and the rest for metallurgical use. NNMS, JNMS and ZCCW are the main producers of tantalum wire. However, Baoji Nonferrous Metal Processing Factory, Shanghai Research Institute for Nonferrous Metals and Shenyang Metals Research Institute are the main suppliers of tantalum plates and strips for corrosion-resistant and high-temperature applications, and among these the first plant plays the most important role in China.

Tantalum pentoxide (Ta<sub>2</sub>O<sub>5</sub>) is obtained from K<sub>2</sub>TaF<sub>7</sub> solution through precipitation with ammonia. Ta<sub>2</sub>O<sub>5</sub> is mainly carburized to TaC, which is widely used as an additive in the cemented carbide industry. Recycled tantalum scrap and powder are increasingly used as raw materials for TaC production. Although mixed carbide (Ta.Nb)C is used in some cases, straight TaC is dominant nowadays in China. Particle sizes of most TaC products are coarser than 1.5 µm, but there are some grades with particle size of less than 1.2 µm. Besides the five plants mentioned above, there are some other entities coming into TaC production recently. Among all these, ZCCW is the largest TaC producer, with a capacity accounting for 70% of the national total.

In addition, purified Ta<sub>2</sub>O<sub>5</sub> is provided for production of lithium tantalate crystals (LiTaO<sub>3</sub>), optical glass and ceramic capacitors. At present, there are more than 20 plants and institutes engaged in LiTaO<sub>3</sub> crystal growth in China. So far, the application of LiTaO<sub>3</sub> crystals as surface acoustic wave filters is still less than lithium niobate crystals in China. But due to the unique properties of LiTaO<sub>3</sub>, a prospective market can be expected in China in the near future.

The consumption fractions of tantalum produced in China for different applications in 1995 are given in Table III. It can be seen that the usage in tantalum capacitors dominated the domestic consumption of tantalum. The sum of tantalum consumption

25	%
Capacitor powder	44
Capacitor wire	14
ΤαĊ	29
Mill products	1
Other	3

Table III: Tantalum consumption distribution for different products in China in 1995

including capacitor powder and wire reached 58% of the total consumption. The consumption of TaC was in next position, accounting for 29% of the total.

#### **APPLICATION IN TANTALUM CAPACITORS**

In order to meet the ever increasing demand of both domestic and international tantalum capacitor industries, great efforts have been made by producers in China to improve product quality and expand production capacity in recent years.

#### IMPROVEMENTS IN K2TAF7

Through constant innovation, the production equipment and processing technology of tantalum hydrometallurgy have been renewed year by year, which has resulted in a significant upgrade of K<sub>2</sub>TaF<sub>7</sub> quality and crucial expansion in its output. Great changes have been made to Ningxia Nonferrous Metals Smeltery, as shown in Figures 2 and 3. It can be seen that the impurity content in K<sub>2</sub>TaF<sub>7</sub>, based on the analyses of ten common elements, decreased from 300 ppm in 1981-1991 to 140 ppm in 1992-1995, and will target 80 ppm in 1996. In the meantime, the daily output of K<sub>2</sub>TaF<sub>7</sub> increased from 237 kg in 1981-1991 to 500 kg in 1992-1995 and will target 1400 kg in 1996.

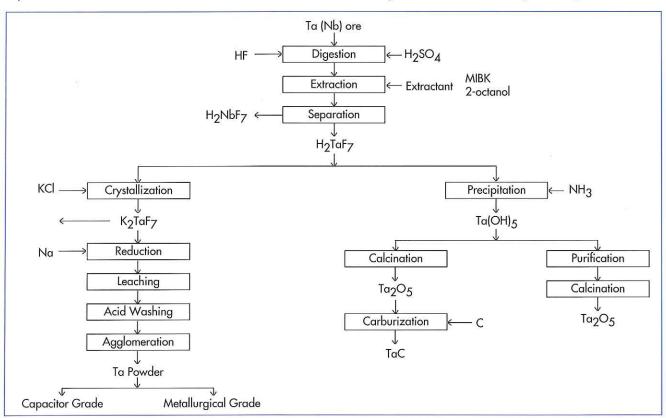


Figure 1: Production flowsheet of main tantalum products

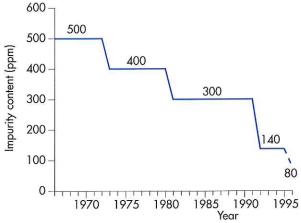


Figure 2: Decrease of impurity content in K<sub>2</sub>TaF<sub>7</sub> produced by NNMS, 1966-1996

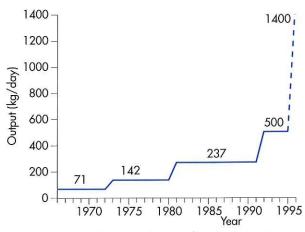


Figure 3: Increase of K<sub>2</sub>TaF<sub>7</sub> daily output of NNMS, 1966 to 1996

#### **ACHIEVEMENTS IN TANTALUM POWDER**

Great achievements in tantalum powder have been obtained through unceasing innovations, such as:

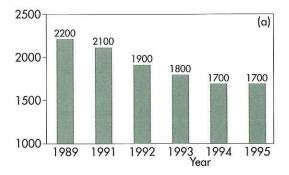
 equipment with better corrosion resistant materials, modern facilities and instruments, many of which were imported from abroad;

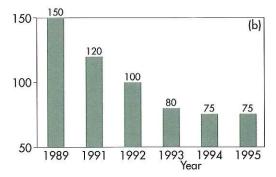
- using high purity raw materials including K<sub>2</sub>TaF<sub>7</sub>, Na, salts, acids, water, etc.;

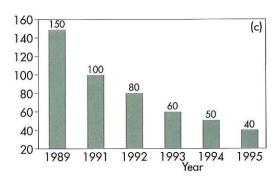
 optimising and accurately controlling technology parameters in every process step, such as temperatures, times, material flows, etc. to make the reactions more complete.

Through these improvements, high specific area and high purity have been realised for capacitor grade tantalum powder. For example, the hydrogen, oxygen, (Fe+Ni+Cr) and (K+Na) contents in FTW230 grade tantalum powder produced by Ningxia Nonferrous Metals Smeltery decreased from 2200, 150, 150, 130 ppm in 1989 to 1700, 75, 40 and 10 ppm in 1195, respectively, as shown in Figure 4.

Depending as they do on high quality, high specific surface area and appropriate physical properties, these kinds of tantalum powders can be compacted into lower densities and sintered at lower temperatures. Thus the available CV values of the powders have been increased significantly year by year, as shown in Figure 5. The typical characteristics of 30 000, 40 000 and 50 000 µFV/g grade tantalum powders produced by NNMS are listed in Table IV. The products with CV values lower than 15 000 µFV/g are mainly used in the home market, while those with CV values higher than 15 000 µFV/g are supplied to customers abroad. Since 1992, the exportation of tantalum powder has increased yearly.







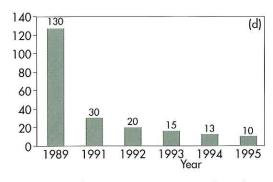


Figure 4: Decrease of impurity content in FTW230 grade tantalum powder produced by NNMS, 1989 to 1995:
(a) oxygen, (b) hydrogen, (c) Fe + Ni + Cr, (d) K + Na, content in ppm.

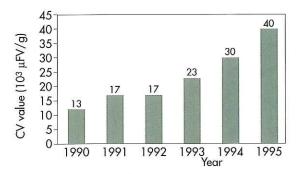


Figure 5: Increase in CV value of tantalum powder produced by NNMS, 1990 to 1995.

Grade	Impurity contents (ppm)					m)		Sintering		CV	Leakage	
	0	С	Fe	Ni	Cr	K	Na	T°C	t min	μFV/g	nA/CV	
FTW300	2400	51	17	14	10	7	<3	1500	30	26610	0.38	
FTW400	2510	50	27	35	5	5	5	1400	30	43900	0.21	
FTW500	3210	83	31	39	5	5	5	1350	30	51996	0.39	

Table IV: Typical characteristics of tantalum capacitor grade powders produced by NNMS.

#### **PROGRESS IN TANTALUM WIRE**

By continuous improvements in tantalum wire production for capacitor applications, much progress has been made in reducing impurity content, upgrading surface finish and improving DC leakage. More importantly, continuous spool to spool production of high quality fine diameter tantalum wire has been carried out at the Ningxia Nonferrous Metals Smeltery since 1993. As shown in Figure 6, the progress in miniaturisation of tantalum wire at NNMS is also very attractive. Before 1986 only 0.4 mm diameter tantalum wire could be produced. In 1987 the production of 0.3 mm wire was realised and soon afterwards, in 1995, 0.2 mm wire began to be supplied to customers. Now, in 1996, even finer sized wire may come on to the market.

The production of tantalum wire has grown rapidly in China in recent years. Also, estimated domestic consumption of the wire in the period 1991 to 1995 increased by three times. Due to the use of high CV powders and fine diameter wires, the miniaturisation of tantalum capacitors was greatly enhanced and as shown in Figure 6 the weight ratio of powder to wire used in tantalum capacitors also decreased dramatically.

#### **FUTURE PROSPECTS**

The total amount of various kinds of capacitors required for the domestic market reached about 50 billion pieces in 1995. There are 273 capacitor producers in China, whose total sales can only meet less than 30% of domestic demand. As to the

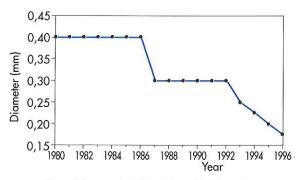


Figure 6: Progress in miniaturisation of tantalum wire produced by NNMS, 1980 to 1996.

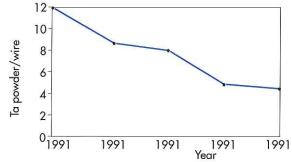


Figure 7: Decrease in ratio by weight of tantalum powder to wire, as used in tantalum capacitors in China, 1991 to 1995.

tantalum capacitor industry which has developed since the mid-1960's, it now includes 11 plants, from which different kinds of liquid and solid types of tantalum capacitors with working voltages in the range of 6.3 to 150 V and working temperatures in the range of -55 to 150° C are produced. Most of these products are widely used in home appliances and commercial electronic devices, such as televisions, video recorders, and the communication, computer and instrument industries. Only a very small portion is produced for military applications.

In the late 1980's, several dip type tantalum capacitor production lines were introduced from abroad. Now the chip type of tantalum capacitor is under development. The output of tantalum capacitors has been doubled in the last three years. But most of the domestic consumption of tantalum capacitors still relies on imports. Tantalum capacitors produced at home took a share of the domestic market amounting to 8, 24.1 and 30.7% in 1993, 1994 and 1995, respectively. A huge market is opening to the worldwide tantalum capacitor industry. It is optimistically expected that the developing tantalum industry in China will see a prosperous future with the incentive of domestic and international demand.

#### **APATITY CONFERENCE ON MOLTEN SALTS**

A Nato Advanced Research Workshop on 'Refractory Metals in Molten Salts - their Chemistry, Electrochemistry and Technology' will be held from August 13th to 18th 1997 in Apatity, Russia, with a large part of the programme devoted to tantalum and niobium. Details may be obtained from Dr D.H. Kerridge, Dept. of Engineering Materials, University of Southampton S017 1BJ, U.K.

#### SYMPOSIUM PROCEEDINGS

The most up-to-date collection of technical papers covering the industry of tantalum and niobium!

Order the book of the Proceedings of the International Symposium held in Goslar in September 1995:

\$US 150 per copy (including postage and packing) from the T.I.C.

#### **MEMBER COMPANY NEWS**

#### Application

We are pleased to welcome an application for membership from King Metallurgical Industry Co., Ltd., Changsha, Hunan, China.

#### Treibacher Industrie

Dr Ulf Stromberger has succeeded Dr Thomas Stenitzer as the delegate to the T.I.C.: Dr Stromberger is the company's new Commercial Director.

#### **Ethiopian Mineral Resources**

Mr Musa Edris, General Manager, is now the delegate of Ethiopian Mineral Development Enterprise (formerly Ethiopian Mineral Resources Development Corporation).

#### Vishay Sprague

In May Dr Felix Zandman, Chairman and Chief Executive Officer of Vishay Intertechnology, Inc., announced that a partnership formed by Vishay and the Eisenberg Group of Companies had signed a Cooperation Agreement with the China National Non-Ferrous Metals Industry Corp. (CNNC), a Chinese government agency. The agreement provides for the comprehensive development of the tantalum industry in the People's Republic of China including the mining and refining of tantalum ore and the production of tantalum capacitors in China through several joint ventures.

Dr Zandman commented that this multifaceted project, when implemented, should lead to an increase in the supply available to current producers of tantalum powder and wire outside China, and supplement the production of tantalum powder and wire in China. In 1995 Vishay made huge purchases of tantalum powder and wire, the primary raw materials used in the manufacture of tantalum capacitors, and this was in large measure the reason for this project which represents an important vertical investment opportunity for the company.

'The parties are currently finishing the definitive terms of the cooperation agreement, which contemplates a separate joint venture for each aspect of the project', added Dr Zandman. 'CNNC has given the Vishay/Eisenberg Group partnership, which will be operated and managed by Vishay, the right to this tantalum project. The first joint venture has as its goal to increase significantly the supply of tantalum ore and raw tantalum concentrate by improving an existing tantalum mine in China.'

The final step of this long-term project, which envisages the establishment of a world class tantalum capacitor manufacturing facility in China, should allow Vishay to continue to expand significantly its presence in China and on the Pacific Rim. The entire project, when fully operational, also calls for the establishment of additional joint ventures to improve the operations of refineries in China.

Dr Zandman continued 'In addition, Vishay anticipates that the cost savings resulting from this project over time are in line with Vishay's continuing effort to concentrate on cutting costs and increasing efficiencies. The savings we hope to achieve through this project, together with other aggressive cost reduction programs the company continues to explore at its existing facilities, such as accelerating the transfer of production to low cost manufacturing countries, should help the company to increase its margins for tantalum capacitors.'

'The joint ventures also intend to explore the development of

other related materials such as lithium, feldspar and niobium which have been found in large quantities along with the tantalum ore.'

#### Vishay Sprague/Kemet Electronics

It was reported in the business press that the Vishay Intertechnology Corporation said that the Kemet Corporation, a rival electronics maker, had rejected its offer to buy Kemet; shares of Kemet rose in Nasdaq trading, while Vishay shares were unchanged. Vishay, based in Malvern, Pa., had been pursuing Kemet, based in Greenville, S.C., since it suggested a friendly merger without specifying a price. Vishay is the largest American maker of passive electronic components including capacitors, and Kemet is the largest American maker of capacitors, continued the report.

#### **Aprobase International**

Associate member Mr Christian Polak, at Aprobase International, promotes the use and development of tantalum and niobium, especially in France (where the T.I.C. currently has no full member company). Mr Polak has written a chapter on the metallurgy of tantalum and niobium, from mines to capacitors, in French, to be published in 'Techniques de l'Ingénieur'. This is widely known among French engineers as an encyclopedia of the industry.

The French Ministry of Industry has given a mandate to Aprobase International to investigate the potential of converting gold 'garimperos' to the exploitation of tantalite-columbite in French Guyana: these gold deposits are rich in Ta-Nb-bearing minerals and could be better exploited industrially.

#### Siemens Matsushita

Siemens installed equipment to increase annual production capacity for tantalum chip capacitors by 80 million capacitors, in chips of sizes B and C. Siemens is constantly making efforts to improve the manufacture and packaging of capacitors and SAW components, such as the introduction of an E case capacitor, 6.3 µF to 330 µF between 4 and 50 V DC. Siemens will also transfer distribution of its 'aktuell' news circulars from 'snail mail' to E-mail and Internet from October 1996 (at which point the T.I.C. will lose touch with it, we suppose).

#### **MR H. SICKENBERG**

Harald Sickenberg, who was for more than 20 years Manager of the European Office of NRC Inc. in Lausanne, Switzerland, before transferring to HCST's Goslar works, passed away in late June of this year.

Harald was well known in the tantalum capacitor industry and will be missed by his many friends.

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