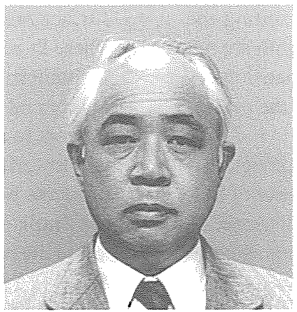
NIPPON STEEL TECHNICAL REPORT No. 85 JANUARY 2002

UDC 669 . 295

Present Status and Future Trends of Research Activities on Titanium Materials in Japan



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Abstract

The special market circumstance and related problems for the titanium industry in Japan are clearly described in this article, by comparing the latest market statistics in Japan and in the USA. In order to solve those problems, research subjects are proposed in the two major areas of corrosion resistant and high specific strength materials. Special attention is also paid to new trends, such as increasing the ratio of titanium alloys, material system separation into two directions and a new widely used alloy development. An aim of this article is to deepen the understanding of the present situation of the titanium industry in Japan and to provide an active discussion on its future direction.

1. Introduction

Japan succeeded in the domestic production of titanium sponge in 1952, a mere four years after the launch of the titanium industry in the United States, and started the industrial production of wrought titanium in 1954. The Japanese titanium industry then developed slowly but steadily, and grew to annually produce 24,000 tons of titanium sponge and 13,000 tons of wrought titanium by the late 1990's.

Titanium research trends in Japan have already been reported by Nishimura[1]. More recently, Moroishi[2] published a detailed report of titanium research in relation to market development. This technology outlook compares the titanium industry structure of Japan with that of the United States as a direct opposite, based on recent statistical data, and clarifies the characteristics[3] of the Japanese titanium industry. The problems facing the Japanese titanium industry are then presented through analysis of its characteristics, and how to solve the problems is proposed from a macro point of view.

The prime purpose of this paper is to present the overall picture of future research issues in the two main areas of corrosion resistant materials and of high specific strength materials with the aid of a grasp of the industrial structure and of a discussion of the relationship between the industry and research trends. If the scope of the presentation is limited to the research trends only in Japan as implied by its title, it may be difficult to understand the overall flow of research on titanium-base materials. Fearing this possibility, emphasis is placed on the results of titanium research amid the flow of titanium research activities the world over.

2. Characteristics and Problems of Titanium Industry in Japan

Table 1 shows the 1998 production and shipment amounts of titanium sponge, ingots, and mill products in Japan. **Table 2**[4] shows the 1998 shipment proportions of commercially pure titanium and

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Table 1 1998 production and shipment of titanium sponge, ingots, and mill products in Japan (statistics of Japan Titanium Society; all figures in tons)

Sponge	Production		24,180	
	Shipment	Total	23,819 (100%)	
		Domestic demand	15,188 (63%)	
		Exports	8,630 (37%)	
Ingots	Production	Total	17,639 (100%)	
		CP titanium	15,463 (83%)	
		Titanium alloys	2,176 (17%)	
Mill products	Shipment by application	Total	12,740 (100%)	
		Domestic demand total	6,146 (48%)	(100%)
		Non-aerospace	4,678 (37%)	(76%)
		Aerospace	567 (4%)	(9%)
		Distributor	901 (7%)	(15%)
		Exports	6,594 (52%)	
	Shipment by type	CP titanium	11,628 (92%)	
		Titanium alloys	1,112 (8%)	

Table 2 1998 percentages by type and shape of titanium materials in United States (RTI survey)

<table>
<tr><td rowspan="3">Percentage by type (%)</td><td rowspan="2">CP titanium</td><td colspan="6">Titanium alloys</td></tr>
<tr><td colspan="2">Near α</td><td colspan="2">α + β</td><td colspan="2">β</td></tr>
<tr><td>All grades</td><td>6242S</td><td>Others</td><td>Ti6-4</td><td>Others</td><td>10-2-3</td><td>Others</td></tr>
<tr><td></td><td>26</td><td>3</td><td>2</td><td>56</td><td>9</td><td>3</td><td>1</td></tr>
<tr><td rowspan="2">Percentage by shape (%)</td><td>Billets</td><td colspan="2">Sheets</td><td colspan="2">Bars</td><td colspan="2">Castings</td></tr>
<tr><td>44</td><td colspan="2">40</td><td colspan="2">14</td><td colspan="2">2</td></tr>
</table>

titanium alloys by type and shape in the United States. Although the shipment data are not shown, the statistics[5] published elsewhere indicate that the 1997 shipment volume of titanium mill products in the United States was 34,200 tons, about 2.5 times greater than that of Japan.

According to the above-mentioned statistics and other related data, the Japanese titanium industry has the following characteristics:

1) Dependence on exports: About one third of titanium sponge production and about a half of wrought titanium production are exported abroad.
2) Overemphasis on commercially pure titanium: Classification by material type of titanium mill products indicates that commercially pure titanium accounts for 92% of the total wrought titanium production and that titanium alloys account for a mere 8%. When titanium mill products are classified by application, 9% are used in the aerospace industry, and the majority are used in the non-aerospace industries.

The U.S. titanium industry may be described as follows:

1) Overemphasis on titanium alloys: Titanium alloys account for 74% of the total production of titanium mill products, and commercially pure titanium accounts for only 26%. Especially, the Ti-6Al-4V alloy occupies 56% of the total U.S. titanium market.
2) Dependence on aerospace applications: Civilian and military aircraft applications account for 75% of the total U.S. titanium market. Furthermore, 75% of that amount is occupied by general-purpose alloys, dominated by the Ti-6Al-4V alloy.

The differences in the titanium industry structure between the two countries basically arise from the difference in the scale of the aerospace industry. The structure of the Japanese titanium industry is opposite to that of the American counterpart, but reports on titanium alloys constitute an overwhelmingly great percentage of the papers published on processing, titanium and titanium alloys in Japan. This discrepancy between the titanium industry and research society is one of the problems for the Japanese titanium industry.

This disagreement is not limited only to titanium, but is common to material development in Japan. For example, when carbon steel accounted for the most of steel products in Japan, steel researchers concentrated on specialty steel and other advanced grades. This discrepancy is put to good use, however, by the development of the high-grade steel manufacturing technology described below. Japan's crude steel production rapidly grew until the middle of the 1970s, but has remained stagnant at about 100 million tons per year since then. The use percentage of high-grade steels, such as IF steel and TMCP steel, is steadily increasing as substitutes for carbon steel in automobiles, shipbuilding and many other industries.

For titanium materials as well, pursuit of a higher alloy ratio is considered to be indispensable from the viewpoint of improving the overemphasis on commercially pure titanium. This proposal was borne of the strong conviction that the present status is not what our predecessors initially envisioned when they inaugurated the titanium industry in Japan. Herein arises the difference between titanium and steel. Basically, commercially pure titanium is a corrosion resistant material, and titanium alloys are high specific strength materials. The increase in the titanium alloy ratio is not of such a nature as to be achieved by the substitution of titanium alloys for commercially pure titanium, but is an issue to be accomplished by expansion of the utilization areas for titanium alloys as high specific strength materials. One concrete measure, although a difficult challenge, is to develop new applications, other than aerospace applications, for titanium alloys as Japan has led the world in commercially pure titanium application development. It is also pointed out that the current single-track strategy of excessive reliance on commercially pure titanium is uncertain as to the maintenance of long-term industrial competitiveness from the standpoints of material technology sophistication and diversification.

Next, the export dependence will have to be maintained for some more time under the present unbalanced supply system that titanium sponge is manufactured only in four countries in the world and wrought titanium materials are produced in a limited number of countries. The presence of trade friction as indicated by the tariff differences between Japan and the United States is certain to pose a problem if Japan supplies good-quality, low-priced products throughout the world. This also reflects the excessive supply of titanium products on the market. There are no concrete measures to counter this situation other than nurturing the sense of thinking about an appropriate product share from a worldwide point of view, as well as promoting the advancement of technology and developing new applications as mentioned above.

In the sections that follow, titanium and titanium alloys will be divided into two main groups of corrosion resistant materials and high specific strength materials, research trends will be outlined, problems will be identified, and future issues will be proposed.

3. Titanium Materials as Corrosion Resistant Materials

The position of titanium as corrosion resistant material as compared with other metals is shown aptly in **Fig. 1**[6], arranged by Stern et al.[7] in 1960 and somewhat supplemented later.

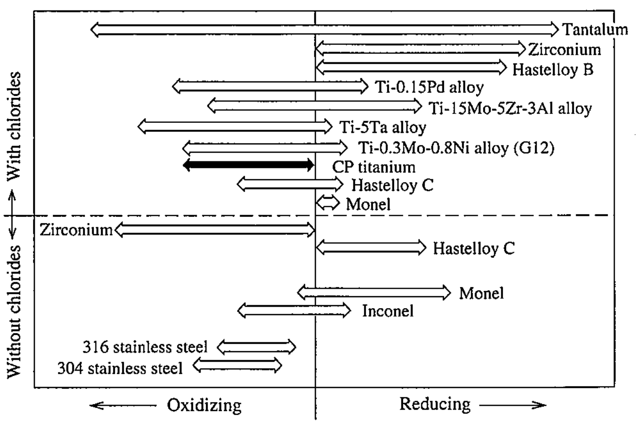


Fig. 1 Corrosion resistance regions of metallic materials

As compared with stainless steel, commercially pure titanium exhibits complete corrosion resistance in an environment similar to that of seawater, and does not suffer pitting corrosion. Since it develops crevice corrosion in a more severe environment, the Ti-0.15Pd alloy was developed. To improve the high-price shortcoming of the Ti-0.15Pd alloy, the Ti-0.3Mo-0.8Ni alloy was developed. In that process, a peculiar corrosion resistance improvement mechanism that depends on the electrochemical characteristics of precipitates was elucidated. Japan developed the three titanium alloys[8-10] shown in **Table 3** as lower-price versions. The technique of adding ruthenium instead of expensive palladium is widespread as well.

Large additions of molybdenum, tungsten, zirconium, niobium, and tantalum were found to be effective in increasing the corrosion resistance of titanium under reducing acid chloride environments. The Ti-15Mo-5Zr-3Al and Ti-15Mo-2.7Nb-3Al-0.2Si alloys[11] were developed for use in such environments. With still higher corrosion resistance expected to be provided by alloys with higher contents of such alloying elements as niobium, tantalum and zirconium (described in the section on biomaterials,) zirconium-base alloys, tantalum-base alloys, and surface treatments every possible measure is considered to have been taken to enhance corrosion resistance of commercially pure titanium.

Another problem is corrosion resistance in the region between commercially pure titanium and stainless steel. Regarding stainless steel, research and development have been actively carried out as represented by seawater resistant stainless steel. Regarding commercially pure titanium, corrosion resistance is sufficient. However, only price is a problem. More efforts will have to be expended to make titanium more advantageous in this competition with stainless steel and to increase the demand for titanium as corrosion resistant material. At present, commercially pure titanium is gradually gaining ground in such fields as civil engineering and building construction, but its excessive quality in such applications is referred to. This reference involves two issues. One issue is the replacement demand for commercially pure titanium as noted for other materials as well. The other issue is the poor initial cost competitiveness of commercially pure titanium because of its high price.

Table 3 Chemical compositions (wt%) of crevice corrosion resistant titanium alloys developed in Japan

Name (Developed by)	Ni	Cr	Co	Pd	Ru	O	N	H	Fe	Ti
TICOREX Nippon Mining & Metals	0.5	-	-	-	0.05	0.05	0.007	0.001	0.03	Balance
SMI-ACE Sumitomo Metal Industries	-	-	0.3	0.05	-	0.20	0.05	0.013	0.20	Balance
AKOT Kobe Steel	0.4	0.15	-	0.01	0.02	0.06	0.003	0.002	0.03	Balance

To lower the price, of course, the use of less expensive raw materials and the adoption of more economical processes are now being tried. The conditions are still unfavorable for commercially pure titanium to compete with stainless steel, however. Naturally, it is not advisable to simply degrade the characteristics of commercially pure titanium in expectation of the renewal demand. Any method whereby the price reduction can coexist with the characteristic reduction merits full discussion and is regarded as a concrete way to counter the excess quality problem.

Many people dislike the above-mentioned method as it detracts from the excellent properties of titanium. Since price and properties are in a contradictory relation for many of the conventional materials, this method does not run counter to economic morality. At the risk of repetition, it may be said that such a method can be applied only when low-price and low-quality products can be manufactured against high-price and high-quality products. One necessary prerequisite is the development of innovative manufacturing technology capable of supplying titanium sponge of variable qualities at lower costs.

The author is particular about this proposal because he regards the above-mentioned research field as mainly concerned with the bipolarization of titanium materials. The need for the bipolarization of titanium materials is strongly advocated to develop new demand in non-aerospace areas, but for what and how the bipolarization should be effected are not concretely clear. This bipolarization is not an issue for high specific strength materials alone, but its possibility should be pursued for corrosion resistant materials as well. The need for the above-mentioned research field will not lead to an easy conclusion, but will be certainly open to further discussion.

4. Titanium Materials as High Specific Strength Materials

4.1 High specific strength alloys for room temperature service

Titanium alloys for use at or near room temperature are increased in strength as shown in **Table 4**[12]. First, alpha alloys were strengthened by addition of sold solution strengthening elements like oxygen and aluminum, but their strengthening was restricted by embrittlement. Then, alpha- beta alloys were developed. Their strength was increased by grain refinement strengthening and transformed beta phase strengthening. Beta alloys were developed from the standpoint of achieving strengthening with the transformed beta phase throughout. Higher strength was attained by making positive use of precipitation strengthening and work strengthening. A maxi-

Table 4 Increasing room temperature strength of titanium alloys

Tensile strength (MPa)	500	1,000	1,500	2,000
Type of alloy	⇐— α alloy —⇒			
		⇐— α + β alloy —⇒		
		⇐— β alloy	—⇒	
Strengthening mechanism	Solid solution	Grain refinement Dual phase	Precipitation	Work

mum strength of 2,000 MPa was achieved in Japan[13]. At that time, the necessity and importance of matrix adjustment were strongly pointed out[13, 14].

Recently, the following equations were proposed to present the effects of alloying element additions on the tensile strength of solid solution strengthened alpha and alpha-beta alloys as the sum of strengthening aluminum and molybdenum equivalents[15].

$$[\mathrm{Al}]^s_{eq} = \%\mathrm{Al} + \%\mathrm{Sn}/2 + \%\mathrm{Zr}/3 + 20[\%\mathrm{O}] + 33[\%\mathrm{N}] + 12[\%\mathrm{C}] + 3.3[\%\mathrm{Si}] \quad (1)$$

$$[\mathrm{Mo}]^s_{eq} = \%\mathrm{Mo} + \%\mathrm{Mn} + \%\mathrm{V}/1.7 + \%\mathrm{Cr}/0.8 + \%\mathrm{Fe}/0.7 + \%\mathrm{Nb}/3.3 \quad (2)$$

$$\rho_u = 235 + 60[\mathrm{Al}]^s_{eq} + 50[\mathrm{Mo}]^s_{eq} \ (\mathrm{MPa}) \quad (3)$$

The strengthening aluminum equivalent equation (1) is characteristic in that the neutral element zirconium and the beta phase former silicon are contained, and indicates that the effects of oxygen and nitrogen are large. These elements have a great strengthening action, but are feared to impair ductility and toughness. Their utilization calls for some ingenuity. Japan has pushed ahead with the development of titanium alloys, such as Ti-Fe-O-N[16], Ti-Al-Fe[17], and Ti-O-Fe-Si[18], from the standpoint of obtaining economical alloys. To grasp the overall picture of titanium cost reduction, a forum carried out research activities for four years and compiled its research results in reports[19,20]. This field is considered to remain an important research area, coupled with the change in the raw material supply system.

In connection with the bipolarization of titanium materials, the author would like to clarify the position of the above titanium alloys containing considerable amounts of interstitial elements. **Table 5** presents the overall picture of titanium material composition proposed by the author. Since oxygen and nitrogen are present in the condition of interstitial solid solutions as known well, the maximum limit to which they can be added without impairing ductility and toughness can be raised. Titanium alloys thus produced cannot become general-purpose alloys for high temperature service, however, because of insufficient heat resistance. It is considered inevitable that new general-purpose titanium alloys demanded from the standpoint of competition with the Ti-6Al-4V alloy should be divided into two classes of alloys in terms of service temperature. Table 5 is still insufficient, but presents basic ideas for the bipolarization of titanium materials and important issues for future discussion.

As already mentioned, high specific strength materials are often used in such environments that special properties are required in addition to room temperature strength. These properties are termed additional properties, and typical examples are given in **Table 6**[12] as basic alloy systems and improvements made to them. The alloy systems described in Table 6 are basic systems comprising the research results accumulated over 50 years. Alloying elements are added to these basic alloy systems to complete individual alloy systems. Since corrosion resistance is already discussed, corrosion resistant materials are included in Table 6, but are omitted from any discussion herein.

Table 5 Overall picture of titanium materials and position of next-generation general-purpose titanium alloys

	General-purpose materials	Special materials
Corrosion resistant materials	CP titanium	Corrosion resistant alloy
High specific strength materials		
Aerospace application	Ti-6Al-4V alloy	Heat resistant alloy
Non-aerospace application	High oxygen α and α + β alloys Next-generation general-purpose alloy	Heat resistant and wear resistant alloy

Table 6 Trends in additional property improvements of high specific strength titanium alloys

Additional property	Basic alloy system	Improvement
Heat resistance	Ti-Al-Si Ti-rare earth element oxide, boride	Oxidation resistance, creep strength Service temperature rise
Corrosion resistance	Ti-Pd, Ru Ti-Mo-Ta-Nb	Crevice corrosion resistance Non-oxidizing environment corrosion resistance
Wear resistance	Ti-TiC Ti-TiB	Particle-dispersed structure control
Biocompatibility	Ti-(Zr)-Nb-Ta β alloying	Toxic element elimination Modulus of elasticity reduction
Workability	β alloying Ti-rare earth element sulfide Mo equivalent limitation	Cold workability Machinability Weldability

4.2 Heat resistant alloys

Improvement in heat resistance and especially the rise in service temperature are strongly demanded as titanium alloys are used as aircraft jet engine materials. With the basic guidelines of improving oxidation resistance by aluminum additions and creep strength by trace silicon additions, the United Kingdom and the United States competed to raise the service temperature of titanium alloys. About 20 years ago, IMI834 was developed in the United Kingdom, Ti-1100 was developed in the United States, and the service temperature reached 600°C[21]. Little research was conducted in this field in Japan.

The adoption[22] of titanium alloys in the engine exhaust valves of mass-production cars in 1998 triggered the research of a heat resistant alloy in Japan. The heat resistant alloy is a titanium-base metal matrix composite (MMC) made by a powder metallurgy process having a dispersion of TiB particles. As suggested by this example, the heat resistance improvement by composition control is saturated at the temperature of about 600°C as achieved with the above-mentioned two alloys. Strengthening by the combination of precipitates or dispersoids with the matrix composition is expected to further enhance the heat resistance of alloys[23]. Particularly, research efforts are concentrated on rare earth element oxides[24] and carbides in China and on borides in Japan.

Development of MMCs that combine matrix solid solution strengthening and dispersed particle strengthening is an urgent issue for heat resistant alloy research. A further rise in the service temperature is expected to be transferred to intermetallic compounds and fiber-reinforced composites.

4.3 Wear resistant alloys

The largest disadvantage of titanium alloys is its poor wear resistance and the resultant restriction of its application to the sliding surfaces of machine parts. To overcome this shortcoming, many types of surface treatments are attempted. Methods are also tried to impart wear resistance to titanium alloys themselves.

Two basic ideas involved are the combination of hard dispersed particles and relatively soft matrix, and the improvement of the interface adhesion between the particles and matrix. Two methods are under study in Japan. One method is concerned with the addition of chromium carbide to the molten metal of the Ti-6Al-4V alloy and the resultant dispersion of TiC in the beta phase matrix[25]. The other method is designed for the internal generation and uniform dispersion of TiB in the sintering step of the powder metallurgy process26). Each method improves the stiffness of the alloy in proportion to the volume fraction of particles, and the latter method can increase the heat resistance of the alloy in combination with the matrix composition as described in the pervious section. Particle dispersion-reinforced MMCs have great possibilities in this respect27).

4.4 Biomedical alloys

Titanium alloys have higher corrosion resistance in the body fluids than do stainless steel and cobalt-chromium alloys, and are gradually spreading as biomaterials. Of the titanium alloys, the general-purpose alloy Ti-6-Al-4V is widely used, but the toxicity of the vanadium contained therein is feared. Development of new biomedical alloys is being carried out in two terms of eliminating toxic elements and reducing the modulus of elasticity to provide stiffness close to that of bones.

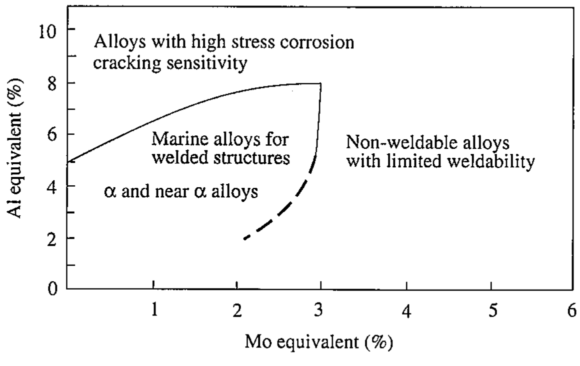
The following two titanium alloys developed in Japan and without aluminum are noteworthy. One is Ti-15Zr-4Nb-4Ta28), an alpha-beta alloy, and the other is Ti-29Nb-13Ta-4.6Zr29, 30), a beta alloy. At present, the Ti-6Al-4V alloy is a mainstream biomedical alloy, and its use is not prohibited yet. From the standpoint of biocompatibility, it is pointed out that we will some day see these new alloys as a mainstream family of biomaterials. To put the excellent biocompatibility of titanium to good use, this research field is expected to become more active.

4.5 High workability alloys

Titanium alloys are difficult to work. Particularly, the Ti-6Al-4V alloy is very difficult to cold work. For this reason, many beta alloys were developed31). In Japan, the Ti-15Mo-5Zr-3Al alloy32) was developed in 1970 and highlighted as the first of titanium alloys combining high strength with high corrosion resistance. Development of beta-rich alpha-beta alloys with a higher percentage of the beta phase was sought. The Ti-4.5Al-3V-2Fe-2Mo alloy33) was developed with its workability improved by lowering the temperature at which superplasticity occurs.

To improve machinability, a method34) was developed for simultaneously adding rare earth elements and sulfur together and imparting machinability by sulfide precipitation.

Titanium materials generally have excellent weldability. When there comes the period during which titanium alloys are to be used as large structures under marine environments, the problems of weldability experienced by ferrous materials, such as weld cracking and environmental embrittlement, come to the surface. Europe, the Unites States35) and Russia36) have already entered such a period, and are actively engaged in the development of alloys as shown in **Fig. 2**37) and the reliability evaluation of welds. Japan is still in the stage of using commercially pure titanium under marine environments. This is a research area expected most, in part to increase the percentage of titanium alloys, and is also a research area where the technological and competitive capabilities of the titanium industry are put to test. The author would like to emphasize that it will become an important research field.



$$[\mathrm{Mo}]_{eq} = [\mathrm{Mo}] + \frac{[\mathrm{V}]}{1.5} + \frac{[\mathrm{Nb}]}{3.6} + \frac{[\mathrm{Ta}]}{5.0} + \frac{[\mathrm{W}]}{2.5} + 1.25[\mathrm{Cr}] + 2.5[\mathrm{Fe}] + 1.7[\mathrm{Co}] + 1.25[\mathrm{Ni}]$$

$$[\mathrm{Al}]_{eq} = [\mathrm{Al}] + \frac{[\mathrm{Sn}]}{3} + \frac{[\mathrm{Zr}]}{6} + 10[\mathrm{O_2}] + 16.4[\mathrm{N_2}] + 11.7[\mathrm{C}] + 3.3[\mathrm{Si}]$$

Fig. 2 Titanium alloys for welded structures used under marine environments

5. Conclusions

This article discussed the unique characteristics of the titanium industry in Japan and the discrepancy between the titanium industry and research society, described the titanium research trends, and presented concrete problems facing the Japanese titanium industry. The increase in the ratio of titanium alloys among titanium materials, excess quality, and new trends as represented by titanium material bipolarization and new general-purpose alloys have been discussed in somewhat greater details to stimulate discussions among those concerned. There is one misgiving that the research trends described are not to the process aspects as fully as related to the material aspects of titanium and titanium alloys. Lastly, the author hopes that this article will encourage the readers to deepen their understanding of the situation in which the Japanese titanium industry is placed and to debate more fully the future of the Japanese titanium industry.

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