

Development of a Titanium Material by Utilizing Off-Grade Titanium Sponge

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ABSTRACT

A titanium alloy for forging and a pure titanium material for exhaust systems have been developed for the purpose of reducing the weight of a motorcycle frame and improving the engine performance. A feature of the material is the use of off-grade titanium sponge that includes many impurities for production of titanium ingot. Stable characteristics have been obtained by controlling oxygen equivalent after setting the volume of tolerable impurities by considering mechanical properties and productivity. In spite of low-cost, this material design provided the same design strength compared to the conventional material, and enabled production with existing equipment. Using this new material and changing the manufacturing and surface treatment process enabled us to provide titanium parts at a lower cost. This technology is a fundamental one for producing titanium raw material for motorcycles and automobile applications, and it is expected to apply to other titanium alloys.

1. Introduction

In recent years, strict environmental regulations have been enacted around the world. These regulations are applied to motorcycles as well as automobiles. There is a growing demand for low emission technologies and technologies for improving fuel economy.^{(1) (2)} Therefore, the use of titanium in components has become essential in order to improve motorcycle maneuverability and to improve fuel economy. The high cost of materials has proven to be a major obstacle in the adoption of titanium components in motorcycles. For large motorcycles, there is strong demand to adopt titanium for exhaust system components, such as the exhaust pipe and muffler, which have a major effect on engine output and running stability. However, a large amount of titanium is used in the structural components of the vehicle, the cost of these components must be reduced relative to the cost of the engine components. In previous research conducted on the use of titanium in engine components, a titanium alloy was developed for automobiles by recycling scrap material in a plasma progressive casting furnace (PPCF). This method proved less expensive than earlier methods. Resulting from this work, the world's first titanium connecting rod for a mass-production motorcycle was developed.^{(3) (4)} In order to

reduce the cost of titanium and expand its use, attention turned towards titanium sponge, a raw material which accounts for 1/3 or more of the total cost of producing titanium materials. This effort resulted in the development of less expensive pure titanium and titanium alloys. These materials were later used in an exhaust pipe, a variable control valve device (H-TEV: Honda Titanium Exhaust Valve), mufflers for the 2002 Fire Blade (for Europe), the CBR954RR (for North America and Japan), and exhaust pipe and inlet valves for the CRF450R. Figs. 1 and 2 show external views of some of these components.

2. Development Aims

2.1 Production Process for Wrought Titanium Products

In 1999, almost all of 11,662 tons of wrought titanium products were produced by the Kroll method.⁽⁵⁾ In this method, chlorine and carbon are added to rutile ore, producing titanium tetrachloride (TiCl_4), an intermediate material. After distillation and refining, the material is reduced with magnesium, producing a titanium sponge which is at least 99.995% pure. Next, the titanium sponge is pressed

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Fig. 1 CBR954RR and titanium parts (Muffler, Exhaust pipes, H-TEV)



Fig. 2 CRF450R and titanium parts (Exhaust pipe, Inlet valves)

and shaped into a melting electrode, which is then used to produce a titanium ingot. Subsequently, the ingot is put through the forging and rolling process to produce the final wrought product (sheet, rod, wire, etc.). This long and complicated production process is one reason why wrought titanium is expensive. Fig. 3 shows a comparison of the production process for conventional material and the production process for the newly developed material.

2.2 Current Situation for Off-grade Titanium Sponge

Approximately 27,000 tons of titanium sponge is produced in Japan each year.⁽⁵⁾ However, because there are only a few producers and suppliers of titanium sponge worldwide, the cost is affected greatly by the level of demand for aircraft. Given that titanium sponge of a high level of purity is required for aircraft production, it was necessary to reconsider the titanium sponge specifications for automobiles in order to obtain titanium at a lower, stable cost. Only the

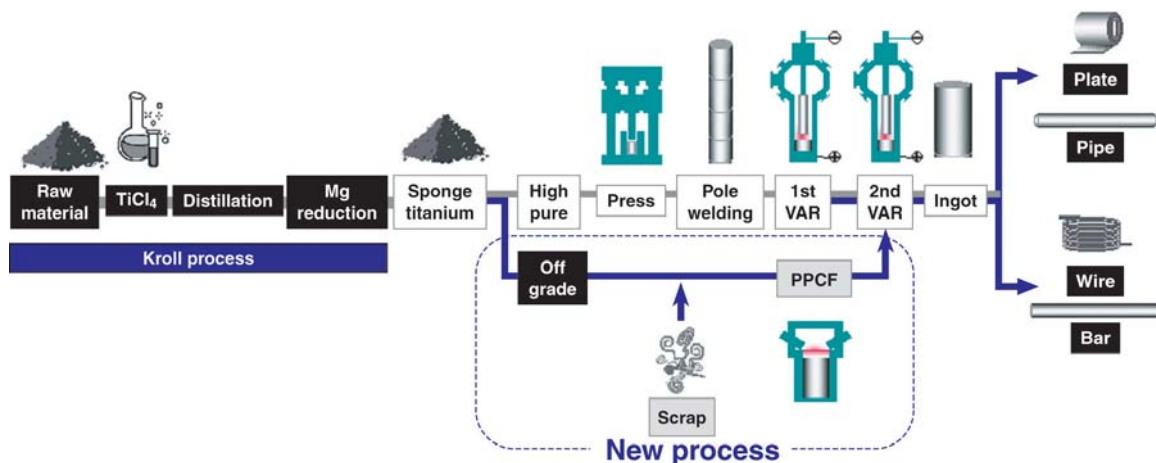


Fig. 3 Manufacturing process of titanium

center of a lump of titanium sponge is pure enough for forging. This pure center accounts for about 80 to 90% of the sponge's weight. The external layer of the sponge lump is known as "off-grade titanium sponge". The sides of the sponge contain Fe, Ni, and Cr impurities that enter the sponge from the stainless steel reaction vessel that is used in the magnesium reducing process, while the bottom of the sponge contains N, Cl, Mg, and other impurities from the chloride reducing process. The off-grade portion is primarily added to steel. When a chemical analysis of off-grade titanium sponge was conducted per JIS H-1610, approximately 60 to 80% of the sponge did not meet MIL or ASTM standards because the iron was too high, otherwise it was of comparatively high purity, as shown in Fig. 4. It was decided to study the possibility of using off-grade titanium sponge for forging.

3. Material Design

3.1 Composition Design of Pure Titanium

The pure titanium that is currently used in motorcycles is primarily used in the exhaust system, namely, the exhaust pipe and the muffler. JIS grade 2 was used, based on strength and processability. These components are primarily shaped by cold pressing and bending. When off-grade titanium sponge is used for shaping, the impurities tend to produce a higher tensile strength and reduced stretchability. Because processing demands strict mechanical properties, impurities can be allowed only to the extent that the material still retains the level of stretchability that is needed for processing.

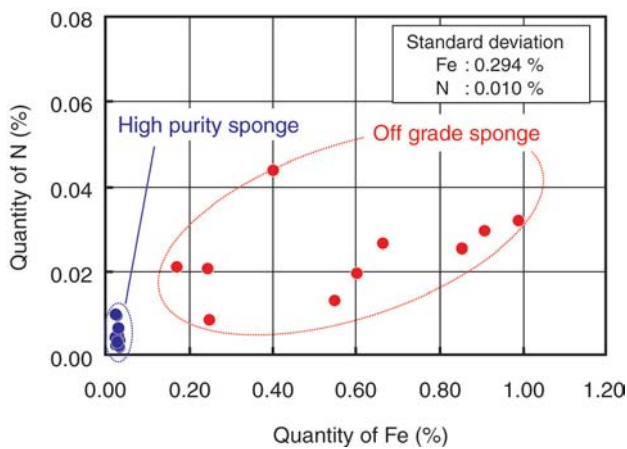


Fig. 4 Distribution of impurities constituent

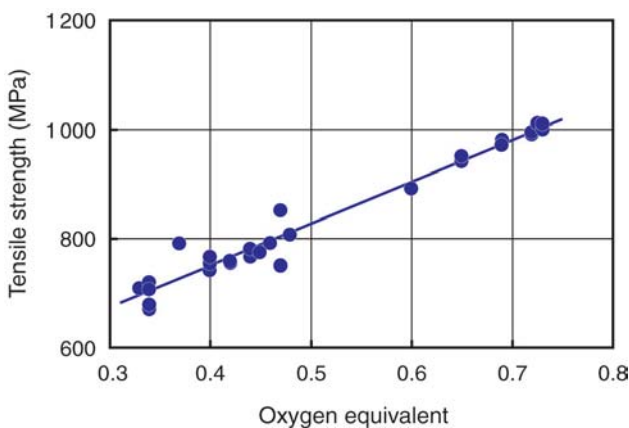


Fig. 5 Relationship between oxygen equivalent and tensile strength

After studying the stretchability needed in various types of shaping processes, 30% was established as the minimum target for stretchability. In addition, while there are many elements that can be found as impurities in off-grade titanium sponge, attention was focused primarily on the elements Fe, O, and N, which have an effect on the mechanical properties. These impurities can be represented in terms of allowable oxygen equivalent as determined by experimental equation (1). The oxygen equivalent is derived by multiplying the amount of each element by a coefficient that reflects that element's effect on mechanical properties relative to oxygen, and then summing the results. The oxygen equivalent is proportional to the mechanical properties. This relationship is illustrated in Fig. 5. Equation (1) shows that the impact of Fe on the mechanical properties is extremely small.

$$\text{Oxygen equivalent } O_{eq} = [O] + 2.77[N] + 0.086[Fe] \quad (1)$$

The allowed amount for each element is decided on the basis of the target oxygen equivalent and the actual distribution of the constituent elements in the titanium sponge. Table 1 shows the stipulated values for the chemical composition of pure titanium developed according to these allowances. Plates produced with this composition are designated as HTPR (Honda pure Titanium Plate Refine), while tubes produced with this composition are designated as HTTR (Honda pure Titanium Tube Refine).

3.2 Composition Design of Titanium Alloys

For reasons concerning strength and availability, the titanium alloy Ti-6Al-4V is frequently used in engine components. Because the mechanical strength of the base alloy is high and the effect from impurities on mechanical strength is low, the service limits for these components compared with pure titanium, could be relaxed. In addition, a high level of stretchability for these components is not required, as they are produced by hot forging.

However, in addition to altering the mechanical properties as described above, the amount of impurities also alters the β transformation temperature, as shown in Equation (2).

Table 1 Chemical composition of HTPR

	wt %			
	H	O	N	Fe
HTPR	0.013 max	0.10 max	0.020 max	0.52 max
JIS TP340-C	0.013 max	0.20 max	0.050 max	0.25 max

Ti: Bal

Table 2 Chemical composition of Ti-64FE

	wt %				
	Al	V	Fe	O	N
Ti-64FE	5.50 ~ 6.75	3.50 ~ 4.50	1.0 max	0.21 max	0.05 max
ASTM Ti - 6Al-4V	5.50 ~ 6.75	3.50 ~ 4.50	0.4 max	0.20 max	0.05 max

Ti: Bal

$$T\beta = 886.0 + 147.7[\text{O}] + 294.3[\text{N}] + 20.4[\text{Al}] - 13.1[\text{V}] - 19.8[\text{Fe}] + \text{K} \quad (2)$$

K: Effect of all other components (C, Mn, etc.)

In order to produce components while maintaining the $\alpha + \beta$ phase, it was necessary to lower the forging temperature below that required for conventional forging. After considering productivity and yield of the forging material, it was decided to reduce the titanium forging temperature by as much as 30 °C compared with the conventional forging temperature. The distribution of constituents in titanium sponge was also taken into consideration when establishing the allowable levels of impurities that would satisfy the forging temperature reduction. Table 2 shows the chemical composition of the developed titanium alloy (Ti-64FE).

4. Results

4.1 Composition

4.1.1 Correlation of composition

In order to obtain the prescribed pure titanium or titanium alloy, titanium sponge and additives containing each alloying element were melted together to form a heat of casting metal. Unlike other metals, it is not possible to control the composition during melting. Instead, a uniform composition is obtained by performing vacuum arc remelt (VAR) twice. In addition, it is not possible to remove impurities other than hydrogen during melting. Therefore, changes in the composition of ingots versus the set composition of off-grade titanium sponge were verified across a number of lots by melting in a mass-production blast furnace. The composition was measured at the top, middle, and bottom of each ingot. The results are shown in Fig. 6. According to these results, the intended composition of the titanium sponge was accurately reflected in the ingots, with no clear segregation of constituents by location.

4.1.2 Low density inclusion

A low density inclusion (LDI) is a localized concentration of an α stabilizing element such as O or N in an ingot, with the characteristic of being extremely hard and brittle. This defect is usually caused by nitrides mixed in the

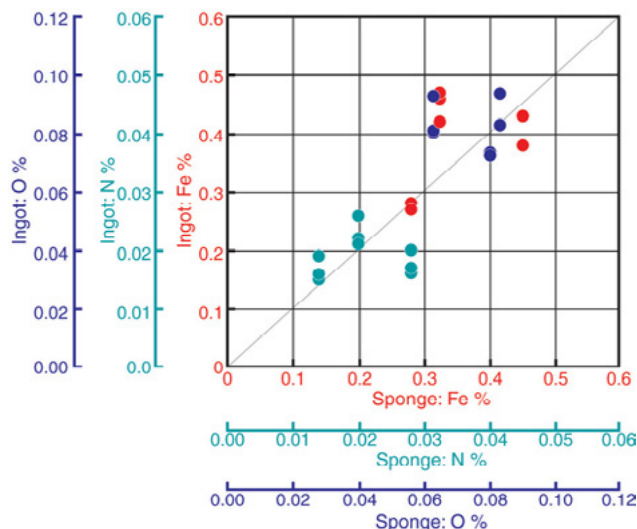


Fig. 6 Comparison with impurities constituent

titanium sponge. Titanium nitrides such as TiN have a high melting point, which poses a concern since they are not completely melted by the VAR process. If using off-grade titanium sponge, it is especially important to verify LDIs in the titanium alloy. This is because they have a large effect on the fatigue strength when the material is used for engine components. In order to check for the occurrence of LDIs resulting from the use of off-grade titanium sponge as a raw material, 800 g ingots produced by prototype melting was evaluated. The three types of titanium sponge indicated below were melted for comparison, and a chemical analysis of the sponge surface was also conducted.

- (1) Titanium sponge suitable for actual use, but at the upper limit of impurities
- (2) Titanium sponge not suitable for actual use due to a high level of impurities
- (3) Titanium sponge of a high level of purity that had been forcibly nitrified.

The results indicated the presence of only two LDIs, both found in sponge (3). No other abnormalities were found. The impurities in the sponge surface consisted almost entirely of iron, with traces of Mg and Cl found adhering to the surface in the case of sponge (2). TiN, the most damaging LDI, was not found.

4.2 Mechanical Properties

4.2.1 Static strength

Table 3 shows the results of tension tests performed on HTPR and Ti-64FE. These tests confirmed the tendency towards increased tensile strength and proof stress and decreased stretchability due to the high oxygen equivalent level compared with conventional materials. The stretchability of HTPR was particularly noted, successfully maintaining at least 30% stretchability at the oxygen equivalent upper limit targeted in the composition design.

4.2.2 Dynamic strength

Pure titanium is generally applied to exhaust system components produced from sheet metal by welding. It is important to evaluate the fatigue strength not only in the base metal, but also in the weld beads and parts exposed to heat. The HTPR fatigue strength evaluation was conducted by means of the Sherk completely reversed plane bending fatigue test. The test was conducted with different flat samples each having different types of TIG welding distinguished by the presence or absence of grooves and filler material. The test results for all samples showed them to be equivalent to JIS grade 2 or better. Fig. 7 shows a comparison of the results for a butt joint which was TIG welded on one side.

Table 3 Mechanical properties

			σ B (MPa)	σ Y (MPa)	EL (%)
Ti alloys	Ti-64FE		1099~1102	1035	15.0~15.6
	Ti-6Al-4V (ASTM SPEC)		895 min	825 min	10 min
CPTi	HTPR ¹⁾	L direction	500~510	333~343	29~30
		C direction	510~519	402~412	33~35
	JIS Grade 2	L direction	353~363	206~216	37~40
		C direction	372~382	235~245	39~40

HTPR¹⁾: Oeq Max condition
L: Rolled direction
C: Rolled perpendicular direction

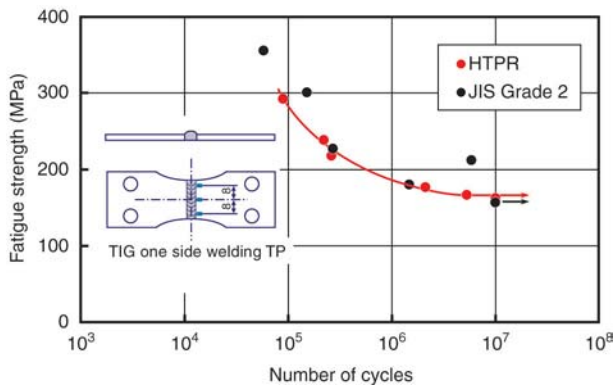


Fig. 7 Fatigue strength of TIG one side welding

Titanium alloys that are used for strength components exhibit a large decline in fatigue strength when notched. As described in section 4.2.1, relaxing the allowable impurity levels tends towards increased strength. This tendency can be enhanced through the oxygen diffusion treatment described in section 4.4. A comparison of the fatigue strength characteristics of Ti-64FE was conducted using the rotating-bending fatigue test. In this test, oxygen was diffused through the air by heating, and the test samples were both plain and notched, with hardened surfaces (OD treatment). The results are shown in Fig. 8. As shown in Table 4, all test samples exhibited fatigue strength that was equivalent to or better than conventional materials. These results indicate that the same strength design criteria that are used for conventional materials can also be used for both of the titanium alloys.

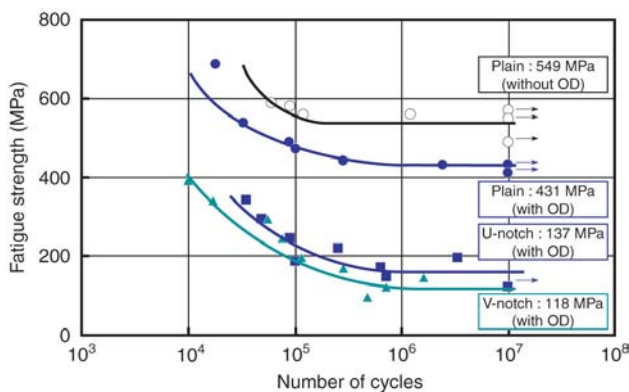


Fig. 8 Fatigue strength of Ti-64FE

Table 4 Comparison of fatigue strength

	Ti-64FE		Ti-6Al-4V	
	with OD	without OD	with OD	without OD
Plain	431	549	323	431
U-Notch	137	314	147	314
V-Notch	118	108	88	118

(MPa)

4.3 Weldability

The test samples created for section 4.2.2 were used to study the cross-sectional structure of the TIG weld and the segregation of impurities. The results are shown in Fig. 9. Cross-sectional observations indicated a tendency towards fineness in the base metal structure in the original HTPR material and also the weld. This is likely due to the effect of Fe, one of the constituent impurities. When constituent segregation of the weld was studied by EDS, no clear segregation of any of the constituents was found. In addition, the weldability of the material was equivalent to that of conventional material under identical welding conditions, with no differences in the amount of material melted or in the extent of the area affected by heat.

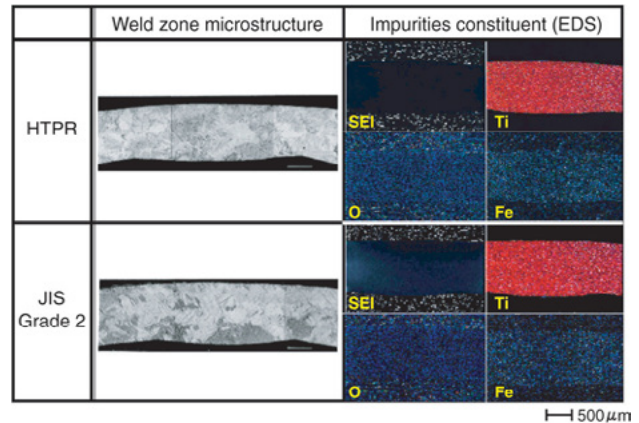


Fig. 9 Weld zone microstructure and impurities constituent

4.4 Durability and Seizure Resistance

Bare titanium metal surfaces exhibit very strong seizure properties. Therefore, the ground surfaces of titanium components must be treated to prevent seizure. Previously, the OD treatment was used to ensure seizure resistance.⁽⁶⁾ In order to check the effect of this treatment on the oxygen diffusion of impurities, a comparison was conducted between the current material and Ti-64FE that had been subjected to the OD treatment. The comparison looked at structure and hardness, and used a faville type adhesive wear test to check the seizure limit load. Fig. 10 shows the results of the faville type adhesive wear test. These results show no difference in the OD treatment structure or hardness, and show a seizure limit load greater than that of the current material. This can be attributed to the original material being harder than the conventional material.

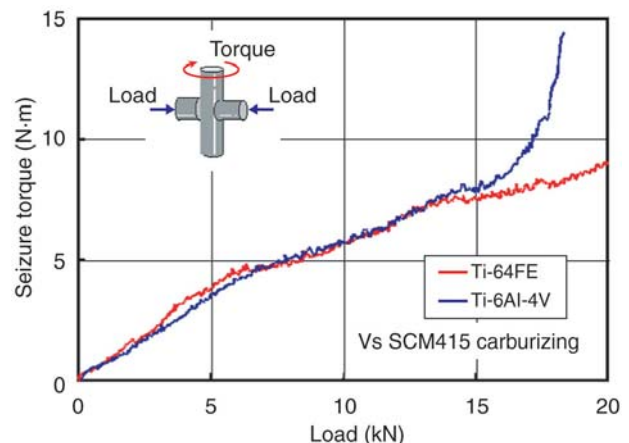


Fig. 10 Seizure property

4.5 Press Formability

In general, titanium cold press formability exhibits different properties from those of steel, as shown in Table 5, due to the small n value (work hardening) and the large r value (anisotropy).⁽⁷⁾ Furthermore, in order to know the limits when using HTPR, test pieces were used to verify the effects on the formability that is necessary in the course of component fabrication. These were tested through a bending test (bending formability), an Erichsen test (punch stretch formability), and an expansion test (stretch flanging). Table 6 shows the results of these tests. HTPR exhibited properties that were roughly midway between those of JIS grade 2 and grade 3, and offered slightly inferior formability (especially bendability) when compared to conventional material. This is related most closely to the stretching value of the mechanical properties. This is due to the fact that although the stretching value of HTPR is set to be equal to JIS grade 2, the effective stretching value of conventional material is higher. When creating components, it is essential that shape design take the actual differences in these properties into consideration.

4.6 Mechanical Formability

In order to use titanium alloys in engine components, it is important to verify differences in mechanical formability in order to consider productivity and costs. In the case of Ti-64FE, as described earlier, increased impurities tend to result in a harder original material. Therefore, a comparison of mechanical formability was conducted on the basis of differences in the time needed to form Ti-64FE under the titanium component forming conditions that have been previously applied in mass production. The machining processes that were checked were milling, drilling, fine boring, and grinding. Data was obtained for each process under the same conditions and for the same processing time as for conventional material, and the results indicated no differences in mechanical formability. In addition, when

Table 5 Property of the formability of titanium

1) Small $\sigma_{0.2} / E$	Poor	Large spring back
2) Hard to carry out work hardening. (n Value)	Good	Easy sharp forming
	Poor	Low limit of forming
		Easy to come out of sectional deformation
3) Large anisotropic (r Value)	Good	Excels as compared with steel sheets (deep drawing)
	Poor	Large radius of bend

Table 6 Formability of HTPR

	n Value		r Value		Erichsen cupping test	Guided bend test	Punch stretch test
	L direction	C direction	L direction	C direction	Erichsen Number	Radius of bend	$\phi 12$ Hole expansion
HTPR	0.131	0.085	2.3	5.6	8.5	1.5t	83%
JIS Grade 1	0.141	0.074	1.9	9.9	13.0	Close contact	165%
JIS Grade 2	0.135	0.089	2.5	6.3	10.5	Close contact	140%
JIS Grade 4	0.100	0.083	1.0	5.5	5.7	2.5t	64%
SUS304	0.470	0.511	1.0	1.0	—	—	—

L: Rolled direction

C: Rolled perpendicular direction

these conditions were re-examined under changed conditions, such as replacing cemented carbide tools with throw away inserts, optimizing the processing conditions (cutting speed and feed), and adding coatings to tools, it was found that the processing time could be reduced by half compared to conventional materials.

5. Component Production Technologies

5.1 Muffler

Regarding independent forming factor evaluation during cold press forming described in section 4.5, each factor is a composite during actual production. When establishing a component press plan, independent forming factor evaluation is inadequate. Therefore, an FLD (Forming Limit Diagram) was established as a map of forming limits.⁽⁸⁾ An FLD is created by scribing a circle on a sheet and then plotting the maximum (piercing) distortion when various mutually perpendicular distortions are applied to the sheet. Based on the growth pattern of the distortion that accompanies progressive forming in locations that are subjected to strong processes, it is possible to predict the risk of fractures occurring, or the degree of safety in avoiding fractures. Fig. 11 shows the FLD for HTPR. These results indicated that, compared with conventional materials, careful planning is essential in the punch stretch forming region.

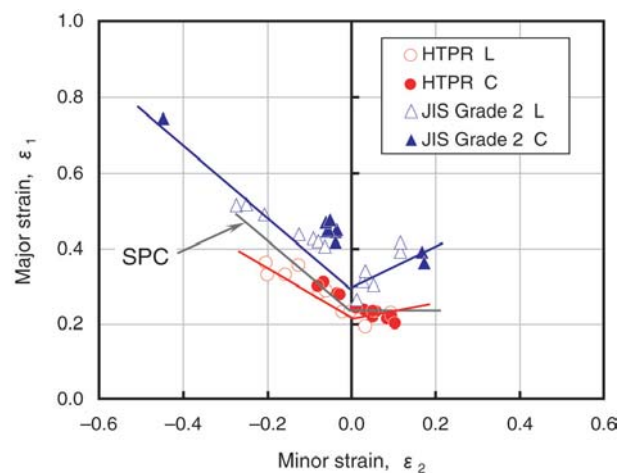


Fig. 11 Forming limit diagram of HTPR

One of the features of the muffler's appearance is the color of the external pipe. Typically, the color of titanium is obtained by some sort of surface treatment such as plating, or by using oxidation interference color obtained by heating in a furnace or by anodizing. By using shot peening on the external pipe, this muffler features a new appearance not found with any other surface treatment.

5.2 Exhaust Pipe

Primarily bending and welding produces exhaust pipes. In the case of the exhaust pipe for the CRF450R where bending a $\phi 41$ -t1.0 pipe into a U shape with R60 (1.5D) was required, the processing was extremely difficult. Application of a mandrel on a drawing and bending bender was necessary. A copper alloy was used for the mandrel material, and the optimal bending conditions were determined, such as the amount to be pushed out, and the force to be used in the rear to push the tube out. In addition to maintaining a flat ratio of 95% or more, the bent shape described previously could be obtained consistently. In addition, it was possible to keep the reduction in the thickness of the sheet on the outer R portion of the bend to less than 10%.

TIG welding was primarily used to join the exhaust pipe components. In light of productivity and cost considerations, however, seam welding was used for the mouth collar joint at the base of the exhaust pipe. In seam welding, the electric current, the weld time, and the pressurization force are important factors. With HTTR, an excellent nugget could be obtained under the same conditions as JIS grade 2.

5.3 Inlet Valve

Table 7 shows the specifications for the inlet valve. In order to reduce product costs, Ti-64FE was adopted as the material and the production method for the plain form was altered significantly. Generally, because the valve head is produced by upset forging, wire that has the same diameter as the stem is used for the valve forging material. However, because the production of titanium wire requires a repeating cycle of rolling, wire drawing, annealing, and the removal of the oxidized surface layer, the production yield is poor and the cost is higher than that of rod material. Therefore, extrusion was adopted for the valve material production method, since that method provides valve forging material from inexpensive rod material. Extrusion requires that careful attention be paid to temperature conditions in particular. At low temperatures, the stem may be bent or the head may not

be fully formed. In the vicinity of the β transformation temperature, the stem may fracture. Therefore, the setting for impurity levels and the temperature range in which forming will be stabilized under the forming conditions were discovered. The specifications for the wear resistance treatment called for an inexpensive OD treatment to be applied to the entire surface. In conjunction with this, the pretreatment process was simplified. These changes made it possible to produce less expensive components.

6. Effects on Actual Equipment

Compared with conventional stainless steel equipment, a weight reduction of 400 g for the CRF450R exhaust pipe and of 1.3 kg and 1.2 kg for the CBR954RR exhaust pipe and muffler, respectively, were obtained. These weight reductions contribute to enhanced power characteristics and controllability and stability characteristics. In the inlet valves, the heads are large diameter but at 25 g, the weight of the inlet valves has been reduced by over 40% compared to conventional heat-resistant steel. As a result, it became possible to ensure the stability of the behavior of the valve system in the required range of rpms. It was also deemed possible to reduce the valve spring storage space to the minimum size, and to lower the cam shaft as far as possible. As a result, the increase in the height of the cylinder head was minimized and a lightweight, compact cylinder head was realized. In addition to the effects brought about by these performance improvements, the equipment is also made more attractive by virtue of featuring "titanium-made components."

7. Conclusions

Pure titanium and titanium alloys that are less expensive and not readily affected by fluctuations in market prices since they use off-grade titanium sponge as a raw material, were developed for forged parts.

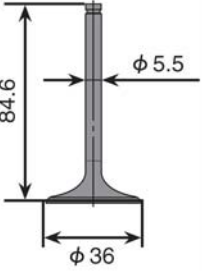
- (1) Verification of the characteristics required by the component production process and management of impurities through oxygen equivalents made the use of off-grade titanium sponge possible.
- (2) The characteristics of this material are equivalent to or better than those of current materials, making it possible to use the same conventional design standards to study components. In addition, because existing facilities can be used for production, no new investments are required.
- (3) By reviewing current component production processes along with the materials, it was possible to supply less expensive titanium components for mass-production vehicles.

This technology is basic to raw materials used for titanium in automobiles. It is expected that it can be widely applied to produce titanium alloys used in other industrial fields.

Acknowledgements

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Table 7 Specification of titanium inlet valve

Material	Ti-64FE	
Stem end	Oxygen diffusion	
Stem	Oxygen diffusion	
Face	Oxygen diffusion	

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