

Production of Gas Atomized Ti Alloy Powder by Levitation Melting Furnace with Electro Magnetic Nozzle

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It is difficult to produce titanium alloy powders with low oxygen content because of the high reactivity of molten titanium against to refractory material and atmospheric air. CCLM (Cold Crucible Levitation Melting) furnace supplies high temperature and large volume of molten titanium without increasing impurities. A new gas atomization process coupled with CCLM furnace and electro magnetic nozzle has been developed to produce high purity titanium alloy powders. Power balance at bottom nozzle, erosion resistivity of the sleeve material to the molten titanium and atomization gas stream have been revealed with experimental study and numerical analysis to construct the process. Spherical and fine titanium powder was obtained through this process and i_s impurities retained almost same level as raw material. It is promising to develop the titanium powder metallurgy products with this process.

Keywords: cold crucible, magnetic field, induction heating and melting, titanium, powder, gas atomization,

1. Introduction

Titanium alloys are being widely used nowadays as they have the advantage of having low density, high corrosion resistance, high specific strength, high heat resistivity and biocompatibility. For example, titanium alloys including TiAl intermetallic compound are using for engine parts of automobile such as connecting rod, turbine wheel and exhaust valve employing their high specific strength and good performance at elevated temperature^{1,2)}.

Because of complicated shape of above products and poor workability of titanium alloy, however, powder metallurgy is expected as one of the production process.

Since titanium is a highly active metal, it pickups oxygen readily during melting and atomizing in commercial production. Too much increase of oxygen makes negative effect to ductility and toughness. Cold crucible levitation melting (CCLM) is well known as a titanium melting process with many advantages such as few contamination, few restriction for material shape and operating atmosphere compare to other conventional titanium melting processes such as VAR and EBM³⁾. Therefore, a new process of manufacturing titanium alloy powders with few impurities expected to construct with utilizing CCLM.

Authors had developed and installed experimental apparatus of cold crucible levitation melting and gas atomization process (CCLM-GA)⁴⁾. However, some problems that caused decrease of the powder yield and the manufacturing efficiency involved in this process.

1. Metal tapping nozzle and furnace bottom made of refractory had to be changed after each atomization.
2. Skull could not reuse after atomization because it expose to atmospheric air at high temperature.
3. Atomization was not stable because of unstable gas stream.

A new system for tapping and atomizing was developed with using the water-cooled copper nozzle and appropriate atomization nozzle design in order to solve above problems.

2. Process Construction

2.1 Melting

Figure 1 shows the principle of cold crucible. High

frequency coil current (J_o) induces magnetic flux (B) and eddy current (J_e) on the surface of the charge metal. Lorentz force generated by J_e and B confines the metal and it keeps uncontact from the water-cooled crucible. The solidified layer of the metal (skull) formed at bottom. Though the Lorentz force is not strong enough to levitate whole metal, still it supports metal confinement without touching to the side wall and keep metal temperature high.

Any shape of material, chipped or massive scrap, is able to melt without downsizing nor performing since the heat source of this process is induction heating. Temperature and chemical composition of the molten metal are very uniform by the electro magnetic stirring. Furthermore, levitation melting does not require vacuum atmosphere such as VAR nor EBM process. This makes it possible to simplify the furnace structure and easy to combine with other devices such as casting and atomization system.

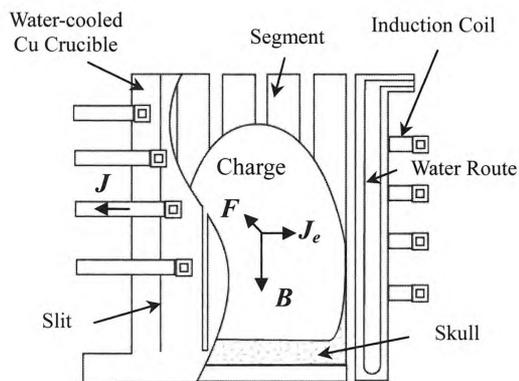


Figure 1. Principle diagram of cold crucible

2.2 Tapping and electro magnetic nozzle

Bottom tapping method has employed for the new process because it does not require tundish and water-cooled cable that has difficulty for high frequency furnace.

Figure 2 shows an experimental apparatus for cold crucible levitation melting gas atomization with electro magnetic nozzle (CCLM-GA-EMN) process. A water-cooled metal tapping nozzle, which is composed of copper segments and coil, is located under a CCLM furnace. Individual coils are wound around the CCLM and metal tapping nozzle and connected to power supplies respectively.

Table 1. Specifications of the cold crucible furnace and electric nozzle

Device	Capacity	Power	Frequency	Coil (mm)	Crucible (mm)
Melting furnace	15kg-Ti	450kW	15kHz	220D×150H×7T	170D×330H
Electro magnetic nozzle		60kW	30kHz	134 ^{top} ~90 ^{bot} D×78H×8T 90D×54H×8T	169 ^{top} ~58 ^{bot} D×50H 58D×18H

2.3 Electro magnetic nozzle design

Electromagnetic levitation forces are described with the equation (1) when eddy current flows near the surface of the conductor^{5, 6}.

$$F = W \sqrt{\frac{\mu}{2\pi f \rho}} \quad (1)$$

F : electromagnetic levitation force [N],

W : consumed power [W]

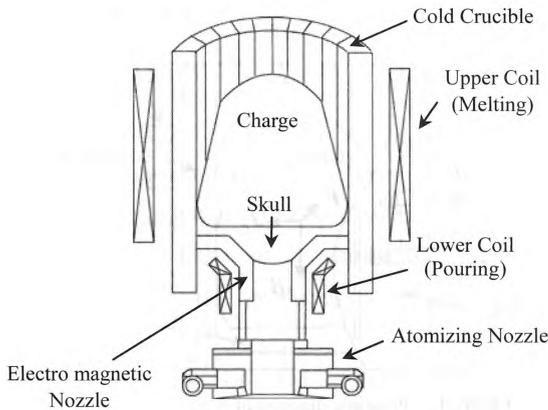
μ : permeability[A/m]

f : frequency[Hz]

ρ : specific resistivity[Ω m]

It is derived from equation (1) that the lower the frequency is, the stronger the electromagnetic levitation force is if consumed power is constant. On the other hand, induced current concentrated at surface leads high heating effect when frequency becomes higher.

Based on above aspects, specifications of the crucible and the electro magnetic nozzle are designed as listed in Table 1. The upper coil (melting furnace) induces strong electromagnetic force for levitating and stirring molten metal and the lower coil (electro magnetic nozzle) supplies heat to metal in nozzle rather than confinement force.

**Figure 2.** Schematic view of CCLM-GA-EMN process

Power distribution of the electro magnetic nozzle system was measured in order to certify the tapping ability. The water-cooled stainless work was set in the nozzle and heated regarding it as charge metal. Energy consumptions at coil, nozzle and work were measured from the temperature rises and flow rate of the cooling water for each device.

Table 2 shows the measured power distributions of the nozzle system. It is revealed that most of the power was consumed by the coil and nozzle consisted of water cooled copper and consequently only 13%, i.e. 8kW, supplied to work. On the other hand, heat loss from metal in the nozzle to tapping nozzle was calculated about 7kW, almost same

Table 2. Power distribution of electro magnetic nozzle

	Power distribution (%)
Work	13
Coil	43
Nozzle	28

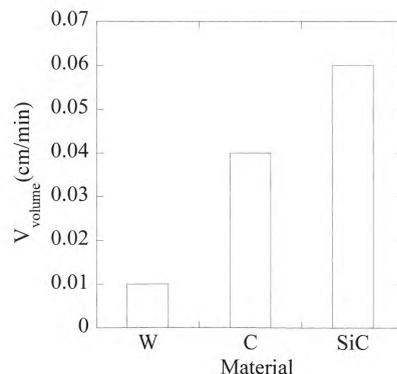
as supplied power, even if the heat transfer coefficient is same order as the reported value of ingot casting process⁷). Contrary to the melting furnace, contact of the metal and nozzle is tight since confinement force act on the metal in the nozzle is not so strong because of higher frequency and metal head.

These results indicated that there is risk for fail in tap when the metal in the nozzle does not melt. Therefore, electrical conductive material (sleeve) was set up in the electro magnetic nozzle in order to supply supplementary heat to the metal in the nozzle.

Material for the sleeve should be selected from the aspects of electrical resistivity, melting point and thermal expansion coefficient. Carbon, tungsten and silicon carbide were chosen as test materials with considering above properties.

Erosion of each material by the molten titanium was measured from dipping test. Cylindrical bars of tested material were immersed in the molten titanium (2023K) for 1 minute. Reduction size of the bars and chemical components of molten titanium after immersion test were analyzed.

Figure 3 shows the erosion rate of each material. Erosion rate of tungsten is the lowest, 0.01cm/min, among tested materials. Erosion affects on atomized powder size since diameter of the powder depends on the metal flow rate. If inner diameter of sleeve is enlarged by erosion, the powder size becomes coarser according to the metal flow rate increase. It requires the sleeve change at each atomization if fine particle is required. Tungsten is the suitable material as sleeve for steady size powder production.

**Figure 3.** Erosion rate of each materials.

2.4 Atomization

The design of an atomizing nozzle is important for stable production of fine powders. Atomization requires a volume deformation work to be done on the metal stream.

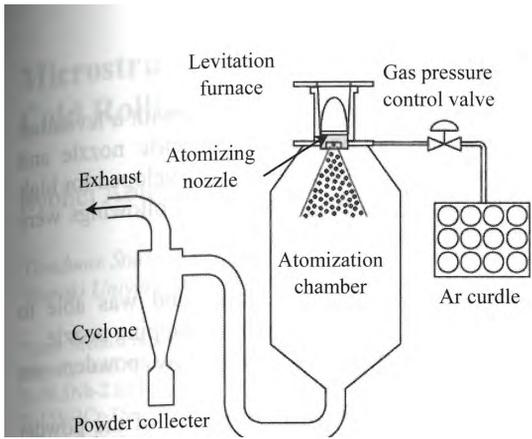


Figure 4. The conventional cold crucible levitation melting and gas atomization apparatus (CCLM-GA process).

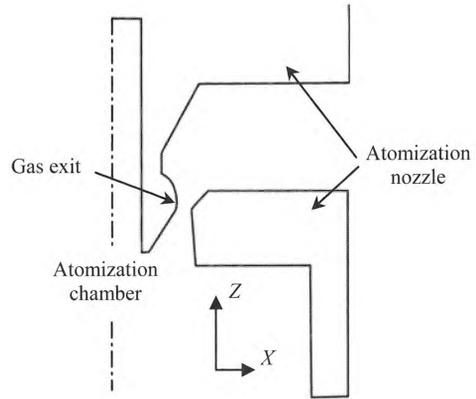


Figure 6. Geometry of simulation model

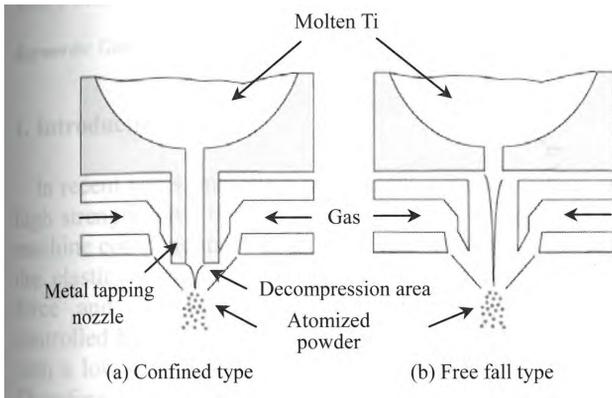


Figure 5. Gas atomization methods

The higher the specific moment (products of gas mass and gas velocity) is, the finer the powders will be (work per melt volume)⁸⁾.

Figure 4 shows a conventional CCLM-GA apparatus. Molten metal exits from the tapping nozzle and atomizing gas ejected from nozzle placed near the metal tapping nozzle to metal stream. Zoom up structure of atomization area is shown in Figure 5. Figure 5(a) shows a confined type atomization nozzle. Metal in the crucible is drawn to the decompression area formed just below the tapping nozzle that is induced by gas stream. Gas exits and the melt stream is approximated with coupling the atomization nozzle to the tapping nozzle. The specific moment of atomizing gas of this system is much higher and the powders become finer.

Problem of this process, however, is that high velocity gas along the tapping nozzle cools protruding part of nozzle and induce nozzle clogging. It is also apprehensive that coil might be attacked by metal stream. Free fall type nozzle shown in Figure 2 and 5(b) is applied for this process from above results. Melt exits from the nozzle under gravity in this system. The atomizing nozzle is designed to atomize the melt stream just above the point of disintegration

2.5 Numerical analysis of gas stream

Gas stream in the different atomizing nozzle geometry were simulated by using a fluid analysis software (FLOW-3D, Flow Science, Inc.) in order to design an appropriate atomizing nozzle setup. Figure 6 shows the 2-dimensional simulation geometry about a full cone type, 360 degree opened, nozzle. The boundary at right side is set the pressure boundary (3.0MPa). Heat and metal stream were not considered. All the fluids were compressive flow and analyzed by state equation.

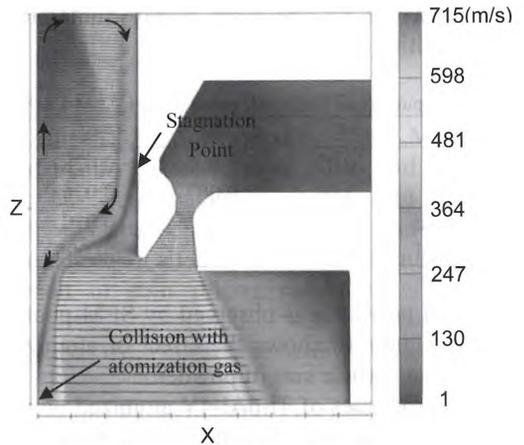


Figure 7. Velocity distribution of the atomization gas in steady-state

Figure 7 shows the calculated velocity distribution of the atomization gas at steady state. The atomization gas move to collision point and then changed its direction to upward. Uprising gas stream circulate along the wall and return back to the collision point. This uprising gas stream induced the unintended powder blowing.

Many semi solid metal powders accumulated at decompression area initiate at the point where the circulated gas broke away from the wall. This phenomenon is caused by forming closed space formed with gas stream film just below tapping nozzle where pressure is lower than atomization chamber.

Pencil type of atomizing nozzle was applied to prevent the pressure difference around the nozzle in order to avoid uprising gas stream. Figure 8 shows a developed atomizing system, CCLM-GA-EMN with pencil type atomizing nozzle.

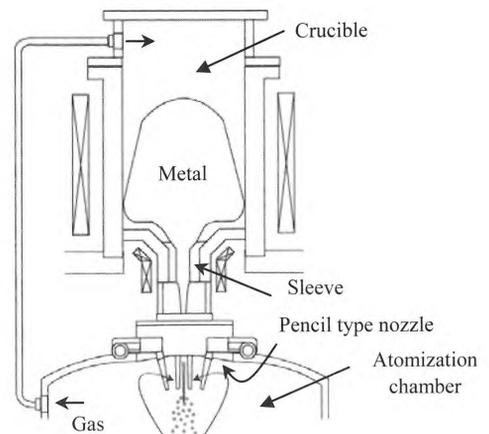


Figure 8. Developed CCLM-GA-EMN process (Pencil type nozzle).

Eight pieces of nozzles attached slanted off vertical were roundly arranged. High pressure gas under the collision point flowed into the decompressed area through gap between the pencil nozzle and equalize the pressures of two areas. In addition, the atomization chamber was connected to the crucible with bypass tube to ensure the pressure balance of three regions. Blowing of the atomization gas was not observed after these modifications and the atomization became stable.

3. Powder Property

Table 3 shows the carbon, nitrogen and oxygen contents of the atomized powders with comparing the powder through CaO crucible and raw material. The powders melted with the CCLM were barely contaminated with these impurities.

Table 3. Impurities of Ti-6Al-4V powders by CCLM-EMN-GA process

Elements	C	W	N	O
#1	0.03	0.005	0.028	0.066
#2	0.03	0.007	0.026	0.057
CaO crucible	0.03	-	0.017	3.6
Raw material	0.01	-	<0.005	0.05

Spherical powder was observed by SEM photographs in Figure 9. Figure 10 shows the effect of atomizing nozzle design on the particle size of powder.

Tensile properties of Ti-6Al-4V atomized powder by the developed process with the sintered test piece produced by Metal Injection Molding (MIM) process have been investigated. All of the test pieces showed good tensile strength (870-900MPa) and is expected as sufficient for products.

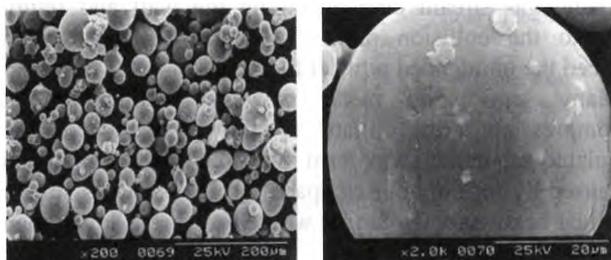


Figure 9. SEM photograph of Ti-6Al-4V powder by levitation melting and gas atomization

4. Conclusions

A new gas atomization process coupled with a levitation, melting furnace and bottom tapping electric nozzle and pencil type atomization nozzle has been developed for high purity titanium alloy powder production. Followings were the confirmed features of the process

- Tapping and atomization was stable and was able to operate continuously without repairing tapping nozzle.
- Pure, fine and spherical Ti-6Al-4V alloy powders are obtained with this process.
- Chemical component and tensile property of the powder showed good performance and is expected as titanium PM products.

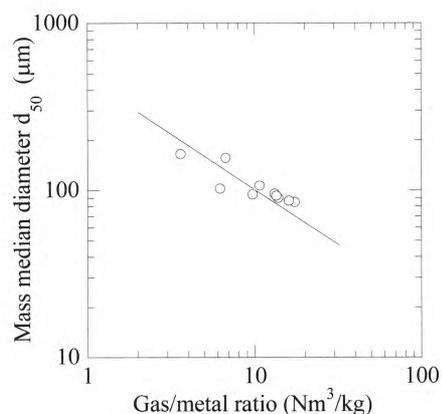


Figure 10. Effect of the gas metal ratio on mass median diameter

REFERENCES

- 1) T. Shibata, N. Demukai and H. Uemura: *Proceedings of the 2nd Electromagnetic Processing of Materials*, (ISIJ, Paris, 1997) Vol.1, pp.231.
- 2) N. Demukai: *Titanium*, 47 (1999) pp.25.
- 3) M. Gamier and I. Madyam: *Proceedings of the 6th Int. Iron and Steel Cong.*, (ISIJ, Nagoya, 1990) Vol.4, pp.260.
- 4) N. Okochi and T. Shimizu, *Denki Seiko*, 74 (2003) pp.227
- 5) E. Fromm and H. Jehn, *Brit J Appl Phys*, 16 (1965) 653
- 6) H. Tadano, K. Kainuma, T. Take, T. Shinokura and S. Hayashi. *Proceedings of the 3rd Electromagnetic Processing of Materials*, (ISIJ, Nagoya, 2000) pp.277
- 7) I. Ohnaka, *Solidification of the steel*, (1977), pp.54
- 8) G. Schulz, *Advances in Powder Metallurgy and Particulate Materials*, 1 (1996) pp.43