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Citation	Transactions of JWRI. 1993, 22(2), p. 249-255		
Version Type	VoR		
URL	https://doi.org/10.18910/8623		
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Effect of Ar or He Pressure on the Formation of Metal Fine Powder by TIG and MIG Arc Melting Process[†]

Yasushi KIKUCHI*, Fukuhisha MATSUDA** and Hiroki IKEDA***

Abstract

Formation process of fine metal powder have been studied by means of arc melting.

High-pressure arc melting method is one of useful process to produce fine metal powder. TIG and MIG arc melting method and pressurized arc atmosphere are used in this experiment. In the paper, results of TIG arc method are mainly reported.

Effect of Ar and He pressure on the formation of fine metal powder are investigated. High-pressure He is more effective to form fine metal powder.

Characterization results of produced fine powder is described.

KEY WORDS: Metal fine powder, TIG melting, High pressure welding

1. Introduction

It is known that metal fine powder has very interesting property which is not observed in its bulk metal. So, the new functional materials using fine metal powders have been studied and developed.

Fine powders of various metal, alloy, ceramic and polymer are required also.

Two types of methods for manufacturing process are commonly used. One is chemical process and another is physical process.

Production by means of Arc melting is one of the useful physical process. It is reported that high purity fine metal powders are produced by physical process, but it is not so good for production efficiency.

Improvements of the fine metal production system by means of Arc melting are needed. The present works have been studied the effect of Ar and He arc atmosphere on production efficiency of fine mild steel powders by means of TIG and MIG Arc melting methods. Also, the effect of Ar or He pressure on the production efficiency was estimated.

Characterizations of produced fine mild steel powder were carried out by X-ray diffraction, SEM and TEM observation. Size distribution of fine powder was determined.

In this paper, the results given by TIG arc melting method are described. On MIG arc melting method, it will be reported next article.

2. Experimental Procedures

Welding apparatus designed to maintain controlled high pressure arc atmosphere was used.¹⁾

A stainless steel (SUS316) chamber of about 1.8 m³ in volume was made to maintain desired gas pressure and composition. The atmospheric pressure in the chamber can be changed from about 15 Pa to 6.5 MPa. The head of an automatic TIG, MIG and sub-marged arc welding machine can be set in this pressure chamber. TIG arc melting was performed with transistorized direct current power source which had an OCV (open circuit voltage) 45V (800A). Drooping characteristics and electrode negative conditions were used. Welding parameters used in this work; electrode: 2%La₂O₂-W diameter 3.2mm, welding current: I = 320A, base metal: mild steel (C 0.13, Si 0.21, Mn 1.13, P 0.019, S 0.006 wt%), base metal moving speed: 0.83 mm/ s, distance from base metal surface to electrode tip; 1a = 1.0mm. In order to collect fine metal powders produced, Al or stainless steel dish (diameter 150mm) was setted to welding touch as shown in Figure 1. Also, dishes were located near the base plate to collect the fine powders falling in atmosphere after arc melting.

The weight of dish was determined at before and after TIG melting.

On a difference of weight, surface area of dish and arcing time basis, total generation rate of fine metal powder during TIG melting was converted.

The chamber was evacuated until under about 133 Pa

+ Received on December 20, 1993

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Transactions of JWRI is published by Welding Research Institute, Osaka University, Ibaraki, Osaka 567, Japan

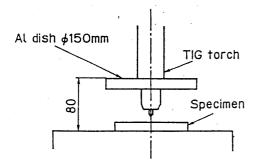


Fig. 1 Arrangement of equipment for TIG.

and then Ar or He gas were introduced to until test pressure, and after arc melting was performed.

3. Experimental results and Discussion

3.1 Formation of fine metal powder by TIG arc in Ar arc atmosphere

Effect of ambient pressure on the generation rate of fine metal powder was determined. Relation between generation rate and ambient pressure of arc atmosphere is shown in **Figure 2**. At 0 MPa (atmospheric pressure), generation rate is minimum approximately 1.6 x 10⁻² mg/s.

This atmospheric pressure shows on the borders of generation rate. It is known that generation rate changes to increase under both vacuum or pressurised arc atmosphere.

The ambient pressure should not be less than 0.5 MPa, generation rate shows in excess of that in vacuo.

Fine powder was analyzed by X-ray method. Result of analysis is shown in **Figure 3**.

Fine powder was identified as a-iron and Fe-oxide (Fe₃O₄). Iron powder was oxidized by residual oxygen in the chamber.

In order to determine the sharp and size distribution of the fine powder, TEM observation was applied.

Figure 4 and 5 show the TEM photographs. It was

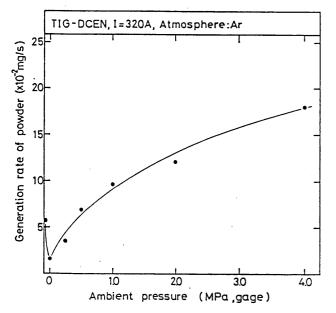
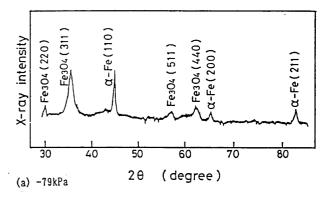


Fig. 2 Effect of ambient pressure on generation rate of powder.



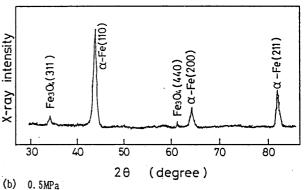


Fig. 3 X-ray diffraction pattern of fine powders.

known that fine iron powder had 2 types of sharpes spherical and polygon. It's size became to slightly bigger increasing with ambient pressure.

In this condition the sizes of fine metal powder are not uniform, its diameter is distributed between from about $5 \sim 6$ nm to hundreds nm.

It is seemed that fine powder has a magnetic property and it is adhering to each other like a chain. Also, in Fig. 5 iron oxide is confirmed by electron diffraction pattern.

Based on the TEM observation results, relation between size, culmulative distribution and ambient pressure was sought.

Figure 6 shows a comparison of culmulative parcentage of powder (particle) and size of powder. Melting conditions are marked up the same figure. The distribution of powder size has a wide range such as several nm to hundreds nm of diameter.

In increasing of pressure, diameter of powder became to slightly bigger.

It is estimated that the fine metal powders are produced by evaporation of metal from molten pool surface on the base plate.

The molten pool is made by TIG arc. So, heat input is important factor on the evaporation. Because, arc voltage is raised remarkably by ambient pressure increase²⁾ and then heat input changes. After all, the heat input increases with pressure raised.

Relation between generation rate of powder and heat input are shown in **Figure 7**. Generation rate is accelerated by pressure increased.

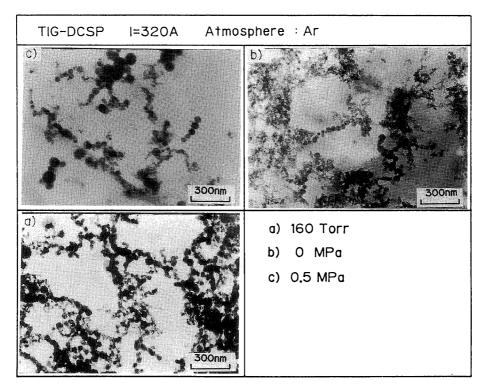


Fig. 4 Morphologies of fine powders by TEM.

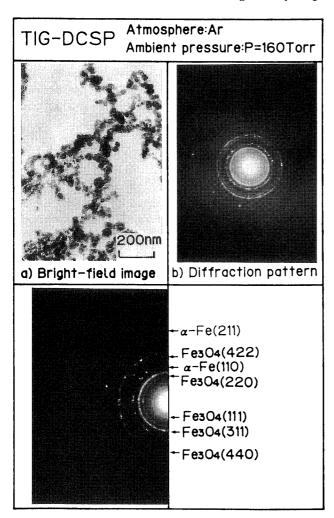


Fig. 5 TEM analysis on fine powder.

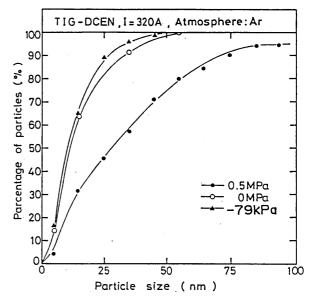


Fig. 6 Cumulative size distribution graph showing comparision of fine particle sizes made by TIG-Ar.

While, the area of molten pool surface varied by heat input changed.

Figure 8 shows effect of ambient pressure on the generation rate of powder as a function of unit molten surface area.

If the pressure of TIG arc atmosphere exceeds about 1.0 MPa, generation rate will be almost constant.

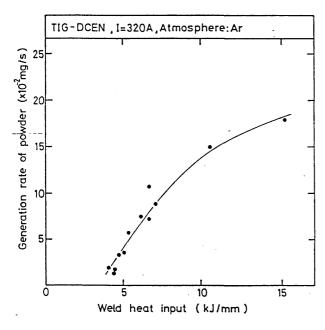


Fig. 7 Effect of weld heat input on generation rate of powder.

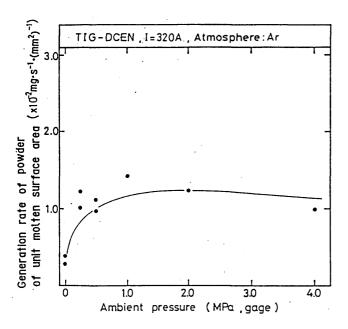


Fig. 8 Effect of ambient pressure on generation rate of powder of unit molten surface area.

3.2 Formation of fine metal powder by TIG arc in He arc atmosphere

The effect of He-gas on the formation of fine metal powder was investigated. He-gas is inert gas as same as Ar, light mass and it is expected to rising the TIG arc temperature. Relation between generation rate and ambient pressure is shown in **Figure 9**. Melting conditions are same as Ar atmosphere, and marked upside the figure. The same tendency of the results in Ar atmosphere is observed. At 0 MPa, generation rate is minimum approximately 1.50x 10⁻¹ mg/s.

In He, this is more efficient in one order as compared with the generation rate in Ar.

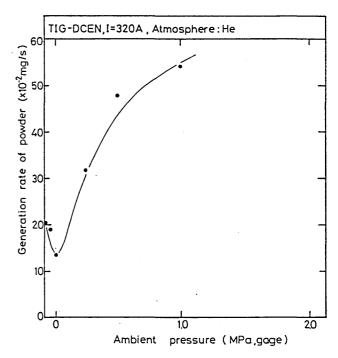


Fig. 9 Effect of ambient pressure on generation rate of powder.

The ambient pressure should not be less than 0.25 MPa, generation rate shows in excess of that in vacuo. Also, in high pressure range, generation rate is more efficient than that in Ar, it shows approximately 3 times. X-ray diffraction was used for the determination of powder component obtained in He atmosphere. **Figure 10** shows analytical result of fine powder. It was identified as a-iron and iron-oxide (Fe $_3$ O $_4$) as same as the powder produced in Ar atmosphere.

Figure 11 shows the TEM photographs. Size of fine iron powder is distributed ununiformly throughout from several nm to hundreds nm. It's size became to slightly bigger increasing with ambient pressure. By analysis of electron diffraction pattern iron oxide is confirmed. Based on the TEM observation results, relation between size, culmulative distribution and ambient pressure was sought. Figure 12 shows a comparison of culmulative parcentage of powder (particle) and size of powder. The distribution of powder size has a wide rang. Also, powder size growth was observed in the ambient pressure raized.

As same as Ar atmosphere, in case of He atmosphere arc voltage raised by ambient pressure increased. Relation between generation rate of powder and heat input is shown in **Figure 13**. The molten pool surface area was spreading by heat input. **Figure 14** shows effect of ambient pressure on the generation rate of fine powder as a function of unit molten surface area.

The same tendency obtained in Fig. 9 was observed. Once a generation rate was decreased until 0 MPa, it was increased with pressure again.

A comparison of Ar and He atmosphere on the generation rate of fine metal powder by TIG arc process is discussed in what follows.

As descrived before in Fig. 5 and Fig. 6, it is clear that

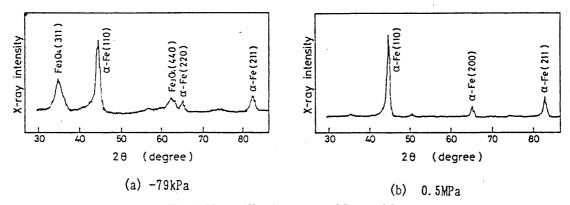


Fig. 10 X-ray diffraction pattern of fine particles.

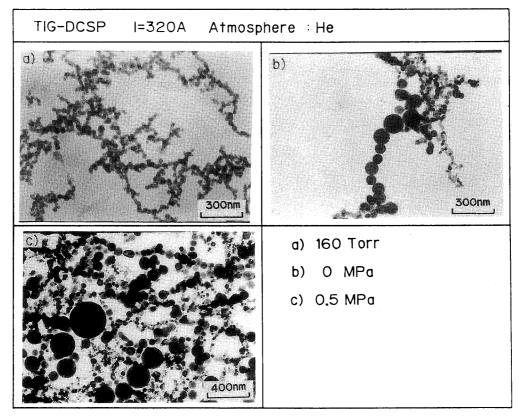


Fig. 11 Morphologies of fine powders by TEM.

He atmosphere is more effective to form fine metal powder. Figure 15 shows relation between arc voltage change and ambient pressure. Arc voltage increased with increasing of pressure. In He atmosphere, higher arc voltage change was observed. Melting current (welding current) was not varied by He or Ar.

Figure 16 shows a comparison of He and Ar heat input change curves as a function of pressure. Heat input was calculated by equation written in the same figure.

Calculated heat input in He atmosphere is much than that of Ar atmosphere. So, molten metal surface area in varied.

Relation between molten metal surface area and ambient pressure are shown in Figure 17. Surface area in He spreads than that in Ar. It is estimated that evaporation of

metal from molten pool surface becomes vigorously.

Temperature of arc is important parameter to formation of fine metal powder.

It is reported that the temperature of arc colum in He atmosphere raise than that of Ar atmosphere.³⁾

In high pressure TIG arc melting, electrode consumption was occured.

In this study, electrode was consumed vigorous in high pressure range. Figure 18 shows electrode appearance after arcing.

In He, electrode was consumed vigorous. Development of new electrode for high pressure use is needed.

The result of good efficiency of He atmosphere can be explained by thermal parameters discribed before.

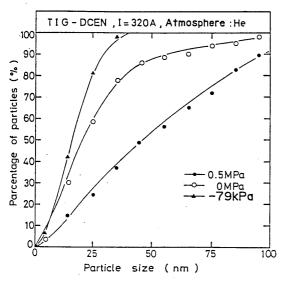


Fig. 12 Cumulative size distribution graph showing comparision of fine particle sizes made by TIG-He.

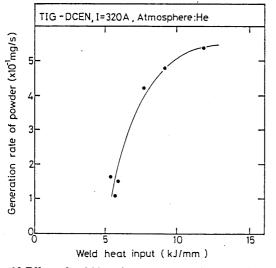


Fig. 13 Effect of weld heat input on generation rate of powder.

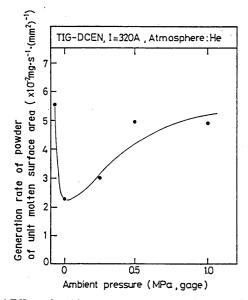


Fig. 14 Effect of ambient pressure on generation rate of powder of unit molten surface area.

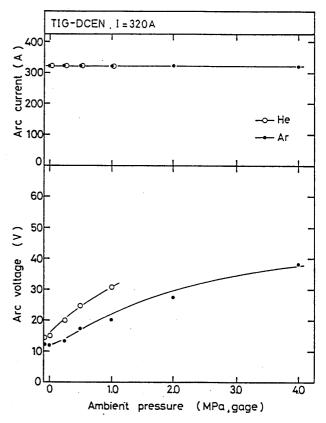


Fig. 15 Effect of ambient pressure on arc voltage and arc current.

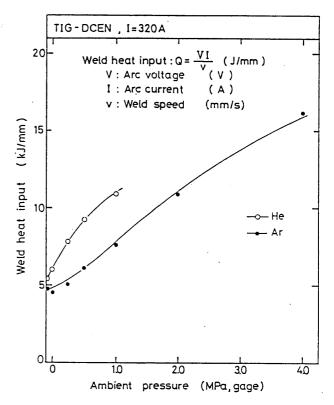


Fig. 16 Relationship between ambient pressure and weld heat input.

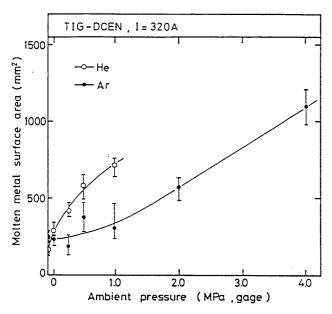


Fig. 17 Effect of ambient pressure on molten metal surface area.

Ambient	TIG-DCEN I=320A	50mm
Pressure (MPa, gage)	Atmosphere:Ar	Atmosphere:He
-0.079 (160Torr)		
0		
0.5	111111111111111111111111111111111111111	6
1.0	шишишиныны	
2.0		

Fig. 18 Electrode tip appearance after melting.

4. Conclusions

- (1) Atmospheric pressure (OMPa) shows on the borders of generation rate of fine powder and generation rate changes to increase under both vacuum or pressurized arc atmosphere.
- (2) He atmospher is more effective to form fine metal powder comper to in Ar atmosphere and generation rate increases with pressure raised.
- (3) Fine powder is identified as a-iron with Fe₃O₄.
- (4) The distribution of powder size has a wide range such as several nm to hundreds nm of diameter.
- (5) Increasing of pressure, diameter of powder becomes to slightly bigger.

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