The Manufacture of Super-fine Tungsten Powder in Plasma

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Abstract. In this paper, the preparation technology of super-fine tungsten powder has been studied by using of induction plasma powder synthesis system, and the flow-ability, particle size, the impurity content of the powder before and after plasma treatment have been measured and compared. The result shows that, by applying for plasma technology ,the powder flow-ability is improved by twenty percent, oxygen and carbon content was decreased markedly, the continuous induction plasma powder synthesis system shows high performance on particle size control.

1 Introduction

The tungsten alloy was an important material for military. Many research focus on improving its properties such as strength and toughness, the research results show that decreasing grain was a best method. The fine tungsten was the best raw material for fine grain tungsten alloy. But the particle size of industrial tungsten powder was 3-5um, it can't be used to manufacture fine tungsten alloy because of too large particle size. A kind of fine tungsten powder should be manufactured, but the finest tungsten powder manufactured in normal method have many impurities content and the approximated particle size was 0.7um. This work targeted to manufacture 10um grain tungsten alloy, the particle size of raw powder should below 0.5 um. A new method should be used to manufacture fine tungsten powder.

Plasma technology was an ideal method for manufactured high melting point powder material, such as : W and Mo. In this process, the raw powder was melting and vaporization because of the plasma's high energy and the powder will become regularly. The plasma manufacture powder technology[1-4] has many advantages, such as :

(1) It can improve the powder's flow-ability[5-6]. The tungsten powder's spherical ratio will reach to 85% after disposed. And the tungsten powder will has a stable performance in hall flow-ability . This change will effectively reduce the powder especially for fine powder segregation and aggregation, and it can effectively control the quality of mixing, loading and pressing process in powder metallurgy.

(2) It can reduce the defects in the powder particles;

(3) It can improve compaction density of the powder;

(4)It can change the powder's morphology, from irregular to sphere in microstructure;

(5) It can reduce the impurities content because of introducing a reducing atmosphere in the preparation process.

In this paper, we can manufacture the tungsten powder using the plasma method, and the tungsten powder's particle size range of 30-500nm. We will analyze and compare the oxygen contents and performance of powder before and after. And we will Study on the influence factors of the plasma method in preparation tungsten powder.

2 Experiments

The figure 1 was schematic diagram of induction plasma powder system.

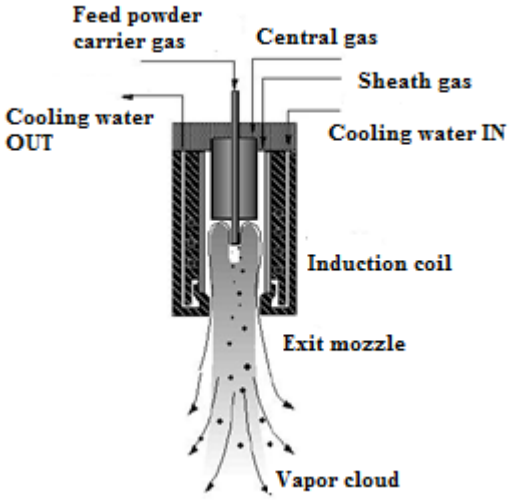

Fig.1 Schematic diagram of induction plasma powder system

The parameters of experiment were shown on the table:

Table 1 The parameters of experiments

Raw material	tungsten powder, which the particle size range in3-5um
Plasma gas	Ar and H2,
Quench gas	Ar
Plasma power	45-60KW
Powder feeding rate	0.5-1.0Kg/h

The tungsten powder was collected from the bottom of the reactor and glove-box. The spherical tungsten powder's properties can be get from testing of laser particle size, SEM, Oxygen content, hall flow-ability.

3 Results and Discussion

3.1 The Hall Flow-Ability

The hall flow-ability between the raw material and finer powder could be compared. And the results were shown in the following table:

Table 2 The flowability testing result

	NO.1			NO.2		
	Raw powder	bottom of the reactor	glove-box	Raw powder	bottom of the reactor	glove-box
Particle size(μm)	3	0.7	0.2	2.6	0.42	0.17
Hall flow-ability	25s/50g	14 s/50g	20s/50g	26s/50g	17s/50g	20s/50g

For the fine powder material, especially the particle size was less than 5 μ m, these type powder have no flow-ability. The results show that the plasma spheroidizing treatment effectively improve

the fluidity of powder, especially for the 1 μ m powder. And there were some changes in the powder's morphology, such as: the powder's surface become regular smooth and dense. And the smooth , dense surface will reduce the friction in the powder, it can improve the flow-ability of the powder.

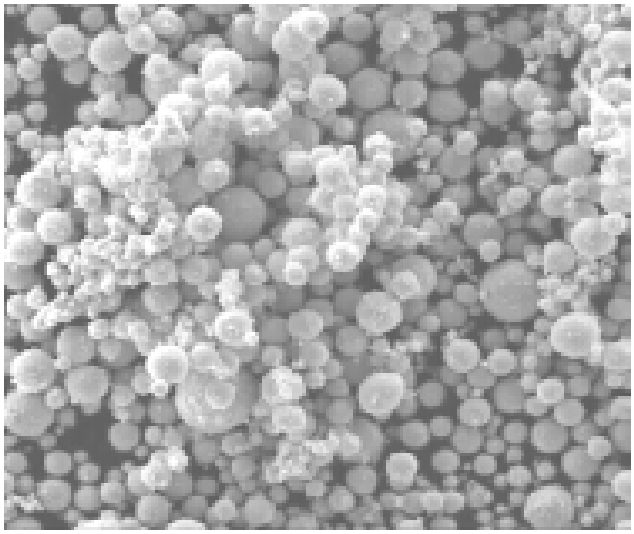

Fig. 2 SEM of treated tungsten powder

3.2 Oxygen Contents

In this experiment, the raw material was two type tungsten powder, which oxygen contents were 0.063%, 0.12% supplied by Xiamen Tungsten CO., Ltd. And the technological parameters of plasma processing was: plasma power were 40 and 60KW, quench gas flow was 1500slpm, plasma gas were argon and hydrogen, powder feeding rate were 0.5 and 1 kg / hour. The results were shown in the following table:

Table3 oxygen content testing result

	No.1			No.2		
	Raw powder	bottom of the reactor	glove-box	Raw powder	bottom of the reactor	glove-box
Particle size(μm)	3	0.497	0.327	3	0.448	0.181
Oxygen content(%)	0.063	0.039	0.051	0.12	0.043	1.03

The results show that the oxygen content of the No.1 was significantly reduced through the plasma treatment. And the oxygen content of the No.2 in glove box was 1.03%, the oxygen content had grown ten times. The reasons of these phenomenon were that:

(1) the superfines powder's activity was improved especially the particle size below 100nm, it can react with air without other condition;

(2) The super fines powder's specific surface area was large, and the oxygen and nitrogen adhere to its surface.

And others data show that the introduction of the reductive atmosphere makes the oxygen deoxidized in the powder manufacture process.

3.3 Particle Size Control

By setting the different test parameters, we can control the powder's particle size level. The results were shown in the following table:

Table 4 Particle size testing result

No.	The parameters	The position of powder	Particle size (nm)
1	Power: 60KW, powder feeding rate:0.5Kg/h	Glove box	38.4
		bottom of the reactor	780.0
2	Quench gas flow: 1200slpm	Glove box	191.0
		bottom of the reactor	448
3	Quench gas flow: 800slpm	Glove box	209.7
		bottom of the reactor	467.1
4	Power: 40KW, powder feeding rate:0.5Kg/h	Glove box	138.0
		bottom of the reactor	890.1
5	H2 flow:50slpm, reactor pressure:95Kpa, Quench gas flow:1500slpm, Power 40KW	Glove box	152.2
		bottom of the reactor	527.2

The results were shown that we can control the particle size through changing the parameter.

(1) Increasing the plasma power can significantly reduce the overall level of the particle size;.

(2) Increasing the quench gas flow can raise the work efficiency;

(3) The addition of hydrogen gas will raise the energy density of the plasma, and it can reduce the levels of particle size.

(4) Decreasing the powder feeding rate can reduce the level of the particle size.

Conclusion

(1) The morphology of superfines tungsten powder was sphere, its powder hall flow-ability was improved 20% than the raw material powder;

(2) The plasma method can reduce the oxygen contents of the powder;

(3) The preparation of superfines tungsten powders can be more precise control of powder particle size of the plasma method.

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