Article ID: 1003 - 6326(2000)03 - 0401 - 04

Specimen preparation of aluminum alloy powders for transmission electron microscopy①

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Abstract: By nickel electroplating at room temperature, the specimen preparation of ultra-sonic gas atomized aluminum alloy powders for observation in transmission electron microscope was carried out. The advantages of this technique are simple technologically and convenient practically. The nickel and the powders combine well in the plated sheet which can be thinned by ion milling. The powders in the thinned sheet possess large thinned area and can be examined in common TEM for the studies of their microstructure.

Key words: aluminum alloys; powders; electroplating; transmission electron microscope **Document code: A**

1 INTRODUCTION

From the study of rapidly solidified powders of different sizes using modern TEM, the information of composition, distribution and crystal structure of metastable phases formed during rapid solidification process can be gotten. Besides, it can provide the theoretical foundation of the optimization of manufacture process and alloy design through the understanding of the microstructure variation with powder size. However, the most rapidly solidified powders equal to and above several microns in size are too large to be electron transparent for common TEM. It is essential for a single rapidly solidified powder to be thinned and polished using some kinds of processing technique. Bendersky et al[1] prepared the TEM specimen of aluminum powders by cold compact, but the deformation may cause pseudo-microstructure. Some other techniques used to prepare specimens of aluminum alloy powders are mainly microtomy[2,3], nickel electroless plating[4], nickel plating powder sedimentation[3,5~7] and copper plating powder adhesion[3].

The microtomy of powders must be conducted on special apparatus[2,3]. It is ideal for fine powders below 5 μm. When the powders are larger than 5 μm, many microtoming slices cured up during the cutting operation and arc trails form in the microtomied specimen due to the quiver of microtoming knife. The specimens are likely to have high dislocation densities and sometimes twinnings are introduced. The microtoming knife is expensive and easy to be damaged. Morra et al[5] developed an electroless plating technique, in which the plating solution possesses a high rate of deposition and a low chemical cost compared with that of an electrolytic nickel plating solution. It is said that the solution may deposit on almost all the material including the container, so it is possible to be used for any kinds of powders. However, the solution which is not stable at room temperature must be stored with refrigeration, even the container must be cleaned according to the standard of analytical chemistry practice. Moreover, the plating solution can not be used repeatedly. A second plating cycle is generally needed when the size of powders is larger than 33 μm.

In the nickel plating powder sedimentation, aluminum powders are added into the plating solution and the solution is stirred constantly so that the powders are co-deposited with nickel on the cathode and enwrapped by nickel[3,5~7]. The solution temperature must be controlled accurately and the excellent plating sheet can be attained only at about 60 ℃. The solution has certain degree of acidity so it will react with the powders mixed. For the TEM specimen preparation of rapidly solidified aluminum alloy powders, this technique is widely used in many cases[3,5~9]. But according to the work of Sheppard et al[7], it is necessary that the interparticle spacing maintains at least 30 μm in the plated sheet so that the powders are not removed from the nickel matrix during the ion milling thinning process. It is quite difficult for the powders deposited on the cathode randomly to maintain certain interparticle spacing, especially for the powders of several microns in size. This certainly decreases the chance of successful preparation of specimens. Furthermore, the observation of

① **Received date:** May 21, 1999; **accepted date:** Nov.24, 1999

the specimens prepared by this technique must be carried out on the 1 000 kV high voltage electron microscope. The copper plating powder adhesion[3] seems to avoid the above disadvantages. The plating is conducted at room temperature and the plated sheet possesses the best ductility and is easy to be thinned. When the technique is used for the specimen preparation of rapidly solidified Al-Cr and Al-Hf powders, ideal results are obtained. However, when the alloys contain many alloying elements and with high hardness, the copper matrix may not be suitable because its relative hardness is lower.

The specimen preparation for TEM should be convenient and be low in cost. In this paper, a specimen preparation technique for TEM observation of aluminum alloy powders using nickel electroplating at room temperature is presented.

2 SPECIMEN PREPARATION

The nickel plating process is conducted with an electroplating device designed by us. It is composed of DC power supply, booster, ampere meter, plating bath and so on. The electrolytic nickel is used as anode and copper foil as cathode. The anode and cathode are settled horizontally and the ultra-sonic gas atomized aluminum alloy powder is placed on the cathode. The schematic diagram of the electroplating apparatus is shown in Fig.1. If needed, the cathode can be divided into several areas in which different sizes or different kinds of powder can be dealt with simultaneously. The chemical composition of the plating solution is listed in Table 1.

The addition of saccharin and dodecyl sodium sulfate as brightener and surface activator is based on the considerations below.

Table 1 The chemical constituents of electroplating solution (g)

$NiSO_4 \cdot 6H_2O$	NaCl	H_3BO_3	Na_2SO_4
130	10	30	40
H_2O	$C_6H_5COSO_2NH_4$		$C_{12}H_{25}NaSO_4$
1 000	0.05 ~ 0.15		0.8 ~ 1.2

1) The cathode is coarse because the powders are placed on it loosely. During the plating process, the solution must have some leveling ability to ensure the nickel depositing faster in the lower parts than in the higher parts, so that the nickel not only enwraps the powders, but also can form a whole strong and ductile sheet.

2) The hydrogen produced on cathode must escape quickly in order to prevent the pinhole forming in the film.

Aluminum is reactive chemically and has a strong affinity with oxygen. A dense oxide film forms on the surface of powder during the atomization process. Besides, some organic matter may contaminate the surfaces of powders in the collection and transfer process. All these factors will reduce the electrical conductivity of powders in the solution and result in the decrease of the bonding strength between powders and plating metal. Therefore, the surface of powders must be preprocessed before electroplating. The preprocess schematic diagram is shown in Fig.2.

The main factors which affect the properties of the electroplating sheet are the pH level of solution and the current density of the cathode. The pH level of the solution should be maintained between 3.8 to 5.1 and the current density of the cathode in the range of $(0.8 \sim 2.0) \times 10^{-2}$ A/cm^2 during the elec-

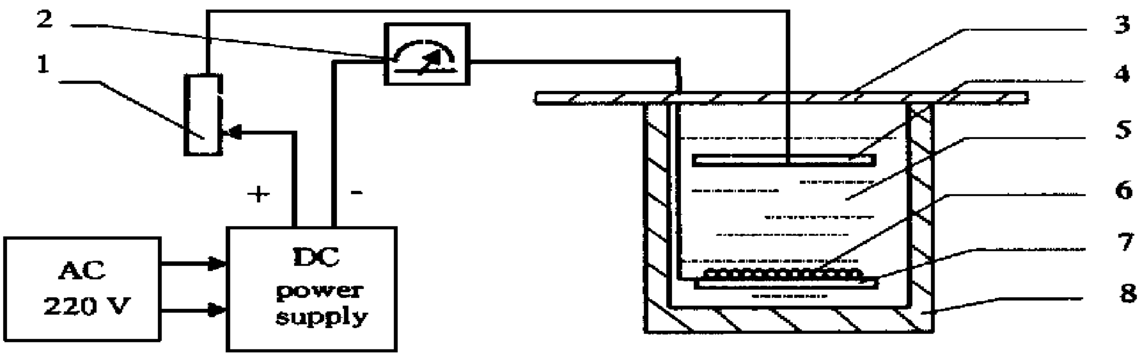

Fig.1 Schematic diagram of electroplating apparatus
1 —Booster; 2 —Ampere meter; 3 —Insulator; 4 —Anode;
5 —Electroplating solution; 6 —Aluminum alloy powders; 7 —Cathode; 8 —Container

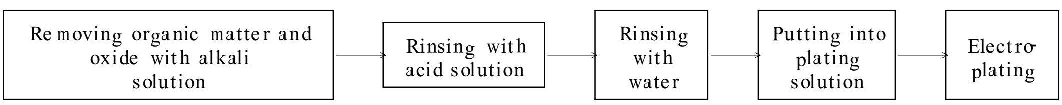

Fig.2 Powder preprocess schematic diagram

troplating process. The electroplating time is controlled by the amount of powder plated each time. When the current density of the cathode is D and the current efficiency is 90 %, here V_0 denotes the occupied volume, V_1 denotes the real volume of the powder treated and S represents the area covered by the powders on the cathode, the electroplating time would be calculated with the following formula[10]:

$$t/\mathrm{h} = 9.03 \times \frac{V_0 - V_1}{SD} \quad (1)$$

After the electroplating is completed, the cathode is taken out of the solution and rinsed in water. The sheet is peeled from the copper foil and mechanically ground to about 30 μm using 800# water resistant abrasive paper. Then it is thinned in an ion milling apparatus with a gun inclination of 20° to perforation and polished with an inclination of 10° for 2 h at the end. The microstructure of the finished specimen is examined in the Philips-EM420 analytical transmission electron microscope with an accelerating voltage of 100 kV.

3 RESULTS AND DISCUSSION

A plated sheet in which the nickel and powders combine well can be obtained by nickel electroplating at room temperature. The uniformly distributed aluminum alloy powders which appear greyish may be seen under an optical microscope after mechanically ground and polished. If the sheet is etched lightly with Killer's agent, it will be found that the powders are enwrapped completely by nickel while the specimen is examined in S-570 SEM (Fig.3).

Fig.4 to Fig.7 show the TEM images of rapidly solidified Al-Fe-Cu-V-Si-Ni-Ce-Zr alloy powders thinned by ion milling. Fig.4 shows a super-fine powder about 2 μm in diameter. The microstructure is of the radiant form. The analysis of electron diffraction pattern indicates that the powder is a single crystal of aluminum. Fig.5 is taken from a powder 10 to 20 μm in size, there are fine precipitates in the cell walls. Fig.6 shows the spherical eutectic of a powder 38 μm to 40 μm in size, and Fig.7 shows the lamellar eutectic microstructure of a powder 50 μm to 61 μm in size.

Compared with the techniques mentioned above, the nickel, in the sheet made by the nickel electroplating at room temperature, not only enwraps aluminum alloy powders completely but combines much better with them during the mechanical grinding and ion milling process. Even in the preparation of powder of 50 μm to 61 μm in size, the powders do not fall off the matrix when the sheet is pre-ground to a thickness about 30 μm and excellent specimen can be gained by ion milling for the study of microstructures (Fig.6). The powders distribute randomly in the plated sheet and there are no restrictions on the inter-

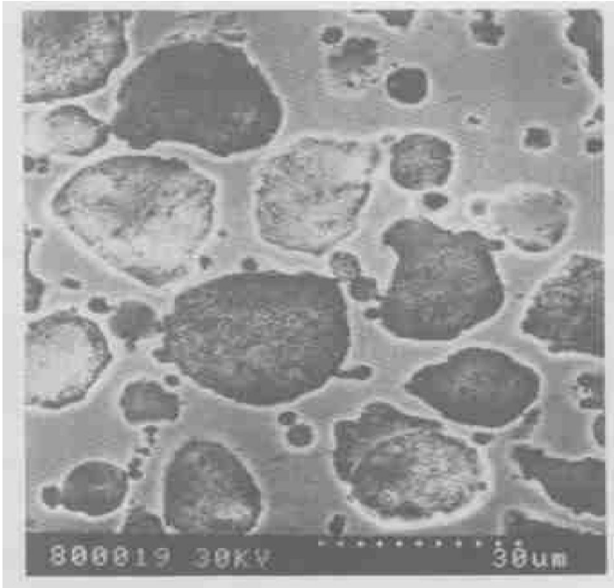

Fig.3 Powders enwrapped completely by deposited nickel in plated sheet

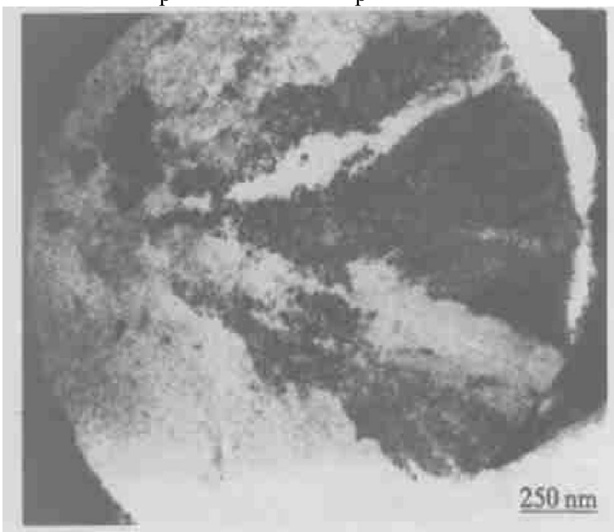

Fig.4 Microstructure with form of radiation from supper-fine powder

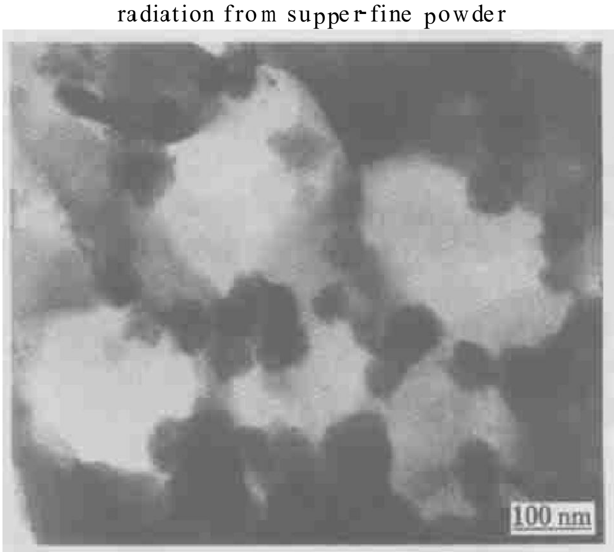

Fig.5 Cellular microstructure of powder 10 μm to 20 μm in size

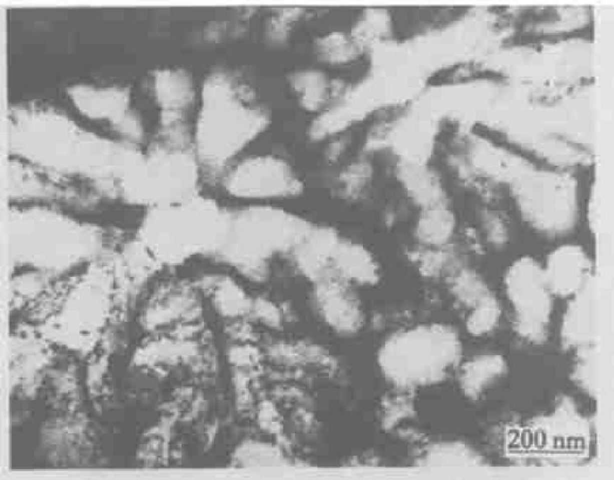

Fig.6 Spherical eutectic microstructure of powder 38 μm to 40 μm in size

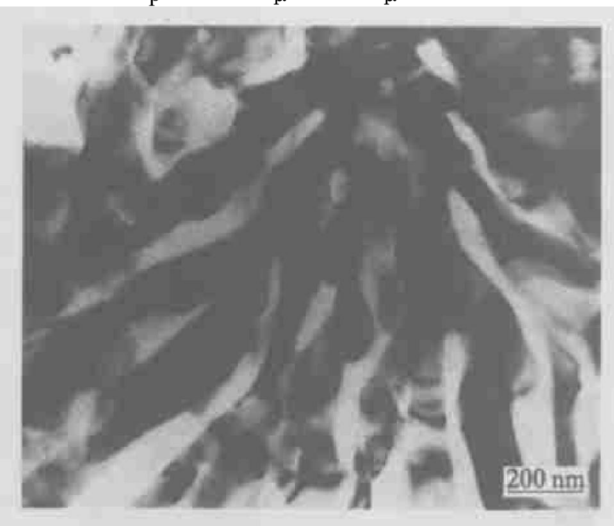

Fig.7 Lamellar eutectic microstructure of powder 50 μm to 61 μm in size

particle spacing (Fig.3). Large thinned area can be obtained in a single powder, especially for fine powder which can be thinned uniformly in the whole cross section (Fig.4). Thus, the specimen, whether it is of fine or coarse powder, can be examined in common TEM. Our technique can be used to treat powders from several to several decades microns in size. With this flexibility, the powders of different sizes can be treated on the same sheet at the same time or treated separately after screened off. Moreover, the plating solution is stable chemically and convenient to store, so it can be used repeatedly. Therefore, the nickel electroplating at room temperature is not only satisfactory to the needs of studies for powders, but also simple technologically. It should be a preferable technique to the specimen preparation of powders for TEM observation.

4 CONCLUSIONS

Under proper preprocessing of the powders and proper control of the experimental condition, nickel plated sheet containing aluminum powders can be obtained by nickel electroplating at room temperature. When the sheet is prepared for TEM observation by pre-grinding and ion milling, large thinned area of powders in the specimen can be obtained. This area can be examined in common TEM for many purposes and it has been proved that the nickel in the sheet enwraps the powders completely and combines well with them. The advantages of our technique are simple technologically and convenient practically. It is a preferable technique to the specimen preparation of powders for TEM observation.

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(Edited by PENG Chao-qun)