Preparation of Amorphous Metallic Powder*

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Synopsis

Preparation of amorphous powders of $Ni_{75}Si_8B_{17}$, $Co_{72.5}Si_{12.5}B_{15}$ and $Fe_{75}Si_{10}B_{15}$ ternary alloys was tried by atomization of their molten metals using a twin roller technique. Atomization was achieved by feeding a stream of molten metal between rapidly rotating rollers with a carbon paste and the disintegrated molten metals were quenched into the water bath. In this paper the critical conditions for atomization were examined using $Ni_{75}Si_8B_{17}$ alloy and the relationship between the volume fraction of amorphous phase and the powder size was clarified. The results suggested that powders with an amorphous single phase might be produced by improvement of this method.

I. Introduction

In recent years there has been growing interest in the production of amorphous metallic powders. The major methods proposed are gas-liquid atomization[1], centrifugal atomization[2], modified melt spinning[3] and electro-spark errosion[4]. Amorphous metals have excellent properties such as soft magnetic properties[5], high corrosion resistance[6] and high strength and toughness[7]. Production of amorphous metallic powders will become a very useful method for applications of their excellent properties.

The works reported herein is to make a trial of preparation of some amorphous metallic powders by atomization using a twin roller technique and to examine the critical conditions for atomization such as a rotational speed of rollers and a gap between rollers and the dependence of particle sizes on the volume fraction of amorphous phase.

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II. Experimental

The mother alloys of $Ni_{75}Si_8B_{17}$, $Co_{72.5}Si_{12.5}B_{15}$ and $Fe_{75}Si_{10}B_{15}$ were prepared by melting mixtures of pure metals, silicon and boron under an argon atmosphere. These compositions were selected in referring Hagiwara's data[8] on the maximum thickness of amorphous ribbons.

The production of powders of these alloys was carried out using a twin-roller quenching apparatus shown in Fig. 1. The apparatus consists of a pair of 80 mm diameter rollers with a face width of 30 mm. The gap between the rollers is set between 0.1-0.3 mm. The rotational speed of rollers can be changed up to 5000 rpm. The mother alloy is melted in a quartz crucible positioned above the rollers by electrical resistance furnace (Siliconit), and then the molten alloy is ejected into the gap between two rollers through a nozzle of 0.3 mm diameter by argon gas of 0.1 MPa.

In order to remain the alloy in the liquid state until disintegration of molten metal occurs between the rollers, the surface of rollers was coated with a carbon paste. After passing through the rollers, the disintegrated liquid particles were quenched into the water bath which was set under 25 mm from the roller nip. Powders obtained were sieved to measure the size distribution and the structure of them was examined by means of X-ray diffractometer, differential scanning calorimeter with a scanning rate of 80K/min and magnetic microbalance.

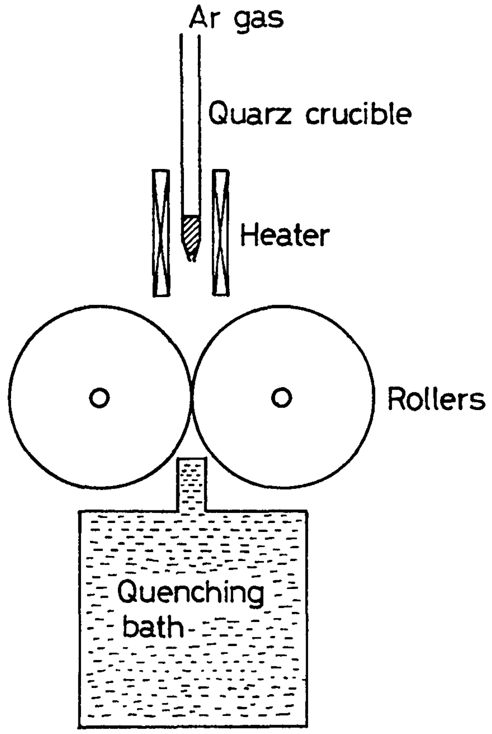


Fig. 1 Schematic diagram of a twin-roller type apparatus used.

III. Results

For roller atomization, the most important point is that the alloy supplied into the gap of rollers remains in liquid state until disintegration occurs completely. For the sake of this settlement, carbon coating on the surface of rollers was found to be suitable. A low thermal conductivity of the coated carbon minimizes the heat transfer to the rollers and also its low wetability against molten metals makes easier to atomize the melts.

Figure 2 shows the critical rotational speed of rollers at which a cavitation in the liquid is induced at various roller gaps. A decrease in the roller gap results in a decrease in the rotational speed of rollers for disintegration of the liquid. Singer et al[9] have suggested that the perforation in the liquid sheet formed between two rapidly rotating rollers is caused by cavitation, in which small holes appear in the liquid sheet and propagate as the liquid sheet moves from the rollers. A mathematical analysis[10] of the pressure distribution between the roller nip indicates that the liquid pressure rises sharply to a large value near the nip between the rollers and drops sharply to minimum just beyond the nip. Before large negative pressures are reached, the liquid is thought to cavitate as a result of expansion of gases contained within the liquid.

The evidences in the present work also supports such a mechanism for cavitation. Figure 3 shows examples that the sample of $Ni_{75}Si_8B_{17}$ alloy is partly perforated at the rotational speed of 100 rpm (a) and is atomized at higher speed of 3000 rpm (b).

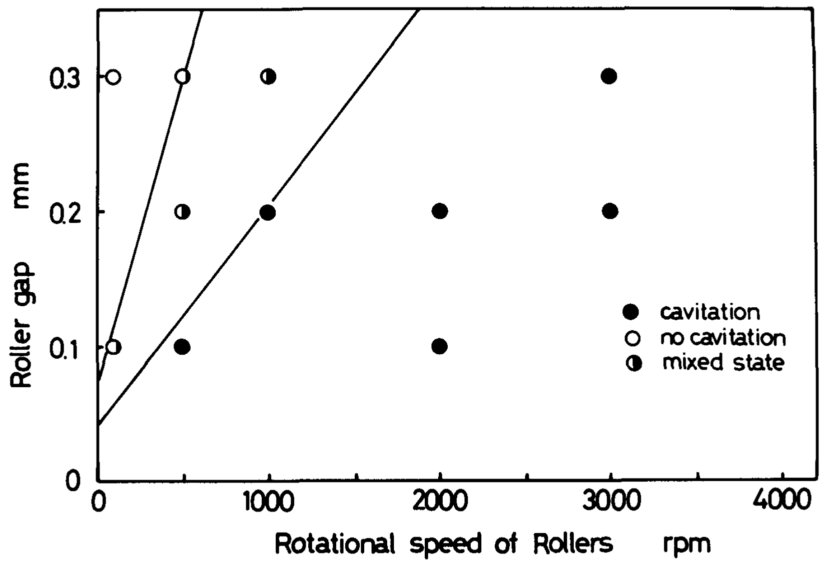
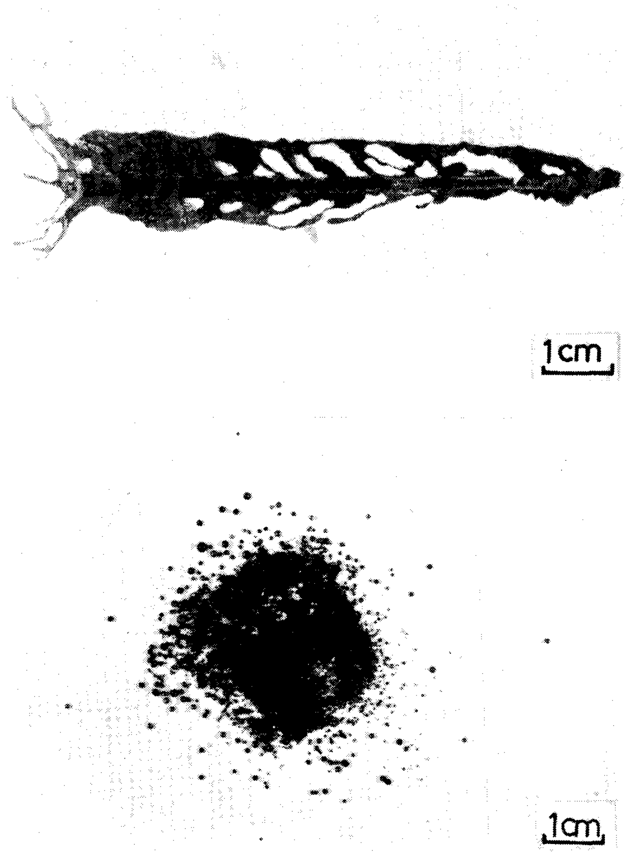


Fig.2 Critical rotational speed of rollers for a cavitation occurence in liguid as a function of the roller gap in the case of $Ni_{75}Si_8B_{17}$ alloy ejected at 1473 K.

(a)

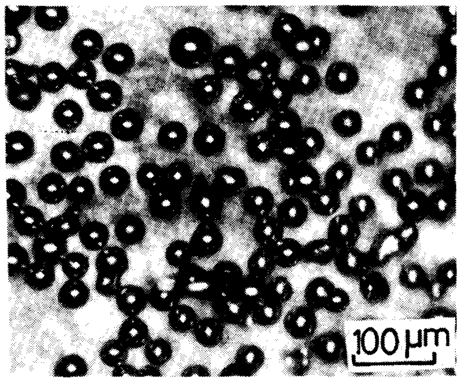


(b)

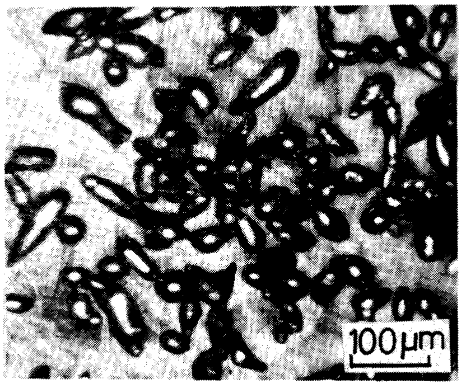
Fig.3 The shape of $Ni_{75}Si_8B_{17}$ alloy produced by a twin-roller apparatus used in the present work.
(a): quenched at the rotational speed of 100 rpm and 0.1 mm roller gap.
(b): quenched at the rotational speed of 3000 rpm and 0.1 mm roller gap.

Figure 4 shows the appearance of $Ni_{75}Si_8B_{17}$ and $Fe_{75}Si_{10}B_{15}$ alloy powders sieved under 400 mesh (37 μm). The powder of Ni based alloy has a spherical shape, while that of Fe based alloy has an irregular and acicular shape. The shape of powders obtained depends on the liquid properties such as wetability and viscosity. The Fe based alloy which is easy to wet with carbon tends to solidify in an irregular form.

After atomization the powders were collected and sieved. The size distribution of powders is represented by plotting the weight percent undersize against the particle diameter. The particle size at 50 weight percent is usually taken as the mean diameter of powders. The particle size distributions of $Ni_{75}Si_8B_{17}$ and $Co_{72.5}Si_{12.5}B_{15}$ powders produced at the rotational speed of 5000 rpm and the ejecting temperature of 1773 K are shown in Figs. 5 and 6 respectively. The mean diameter of powders is about 240 μm for Ni based alloy and about



(a)



(b)

Fig. 4 The shape of $Ni_{75}Si_8B_{17}$ (a) and $Fe_{75}Si_{10}B_{15}$ powders (b) sieved under 400 mesh (37 μm). Both samples were produced under the conditions of 5000 rpm of rotational roller speed , 0.1 mm of roller gap and 1773 K of ejecting temperature.

360 μm for Co based alloy. An increase in the rotational speed of rollers reduces the mean diameter of powders.

The structure of powders was examined by means of differential scanning calorimeter, magnetic microbalance and optical microscope. In Fig. 5 the volume fraction of amorphous phase in $Ni_{75}Si_8B_{17}$ alloy powders quenched in water was estimated by dividing the calorific value by crystallization of the alloy with that of amorphous ribbons of the same alloy produced by a melt-spinning method. An increase in diameter of powders decreases the volume fraction of amorphous phase. Smaller particles are easily quenched into the amorphous state and the powders under 37 μm contain about 85 percent of volume fraction of amorphous phase for Ni based alloy. These results are reconfirmed by observing the microstructure of powders quenched as shown in Fig. 7. Figure 6 also shows the volume fraction of amorphous phase in $Co_{72.5}$-$Si_{12.5}B_{15}$ alloy powders, wherein the fraction was estimated from the ratio of saturation magnetization of powdwes against that of amorphous ribbons at liquid nitrogen temperature. The powders of 37-44 μm in diameter contain about 84 percent volume fraction of amorphous phase.

According to Davies[11], the critical cooling rate for the formation of amorphous phase of $Ni_{75}Si_8B_{17}$ alloy is of the order of $1.1x10^5$ K/s. Therefore, it is assumed that the apparatus used in the present work has the cooling rate of about 10^5 K/s. For this type of apparatus, an important factor to obtain amorphous powders is the distance from the roller nip to the water level in quenching bath as well as the size of atomized liquid. In selecting 25 and 60 mm as this distance, the volume fraction of amorphous phase was examined. Powders produced by a short distance exhibited the volume fraction of amorphous phase higher than that produced by a long distance.

This result suggests that the improvement of quenching method will be necessary for the production of powders with a completley amorphous phase.

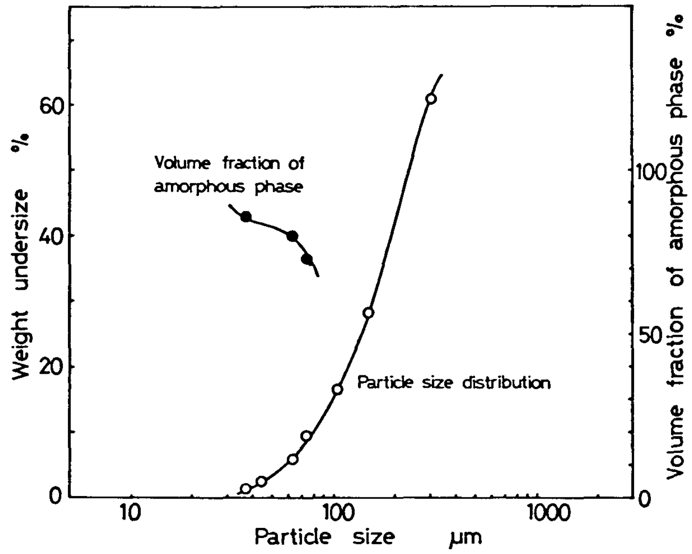


Fig. 5 The relationship between the weight undersize and the particle size for $Ni_{75}Si_8B_{17}$ alloy powders produced at the roller speed of 5000 rpm, the roller gap of 0.1 mm and the ejecting temperature of 1773 K. The volume fraction of amorphous phase is also shown in the figure.

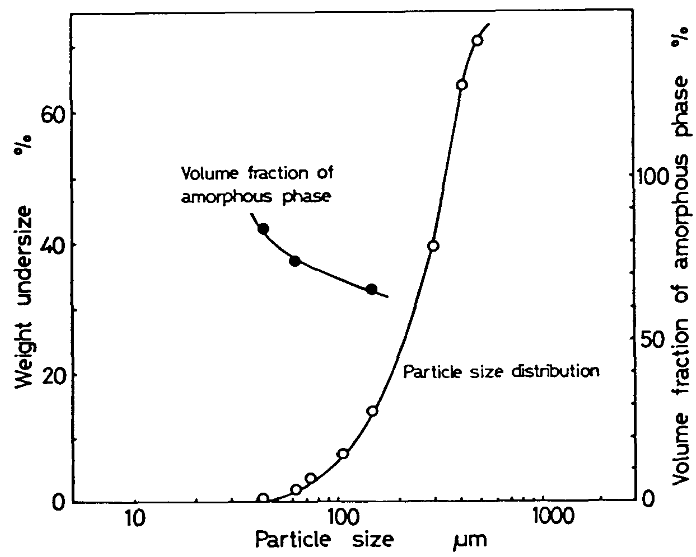
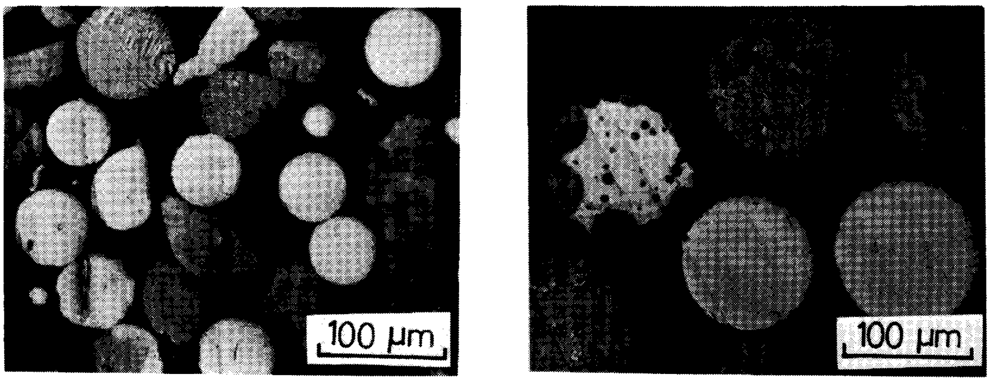


Fig. 6 The relationship between the weight undersize and the particle size for $Co_{72.5}Si_{12.5}B_{15}$ alloy powders produced at the same conditions described in Fig.5. The volume fraction of amorphous phase is also in the figure.



(a) (b)

Fig. 7 Microstructures of $Ni_{75}Si_8B_{17}$ alloy powders with the size of 44-63 μm (a) and 105-149 μm (b), produced at the conditions of 5000 rpm, 0.1 gap and 1623 K of ejecting temperature.

IV. Conclusion

Production of amrphous metallic powders was tried by atomization of the molten metal using a twin-roller technique. Atomization was achieved by feeding a stream of molten metal between rapidly rotating rollers coated with a carbon paste. After passing through the rollers the liquid was disintegrated to small particles under appropriate conditions of the rotational speed, the gap of rollers and the ejecting temperature. A decrease in the roller gap resulted in a decrease in the rotational speed of rollers for the atomization of the liquid. The voume fraction of amorphous phase in powders decreased with increasing the size of powders. The results suggested that powders with an amorphous single phase might be produced by improvement of this method.

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