PRODUCTION OF ULTRAFINE PARTICLES OF OXIDES AND NITRIDES BY LASER BEAMS①

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ABSTRACT

Ultrafine particles of Al_2O_3, MoO_3, TiO_2 and SiO_2 were prepared by irradiate high power laser beams on material surface in O_2 atmosphere as well as TiN in N_2 atmosphere. X-ray diffractometer analysis identified that all oxides and nitrides powder obtained are crystalline except SiO_2 particles which have amorphous structure.

Key words: ultrafine powder, oxide, nitride, laser

1 INTRODUCTION

Modern high technology promotes the development of new materials in different states, such as crystalline, non-crystalline, stable and metastable . In powder metallurgy, materials made from ultrafine particles with diameter smaller than 0.1 μm possesses excellent engineering property. When the ultrafine particles are small enough, a sudden structural change may occur and a new type of materials with special characteristic can be formed. The preparation of ultrafine particles is a major topic in powder metallurgy[1], which attracts a lot of attention in recent years.

Ultrafine powder of pure metal[2] and metastable phases of metal and ceramic[3] have been successfully prepared by high intensity laser beams under Ar atmosphere. In this paper the possibility of forming nm-order ultrafine oxides and nitrides particles by using high intensity laser beams was studied. Their phase analysis was also presented.

2 EXPERIMENTAL METHOD AND PROCEDURES

Pure Al, Mo, Si and Ti6Al4V were used as raw materials for the production of oxide and nitrides. The apparatus and the equipment for laser atomization were the same as those used in reference [3]. 1 atm of O_2 and N_2 were used for laser atomization in the oxides and nitrides formation respectively. Phase analysis, Particle size and distribution were carried out in SIEMENS D500 X-ray diffractometer. Particle size and morphology of the powder were examined by HITACHI-6500 scanning electron microscope and SIEMENS transmission

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electron microscope.

3 RESULTS AND DISCUSSION

The ultrafine particles of oxides and nitrides prepared by irradiating laser beam on materials surface were mostly spherical and elliptical in shape and the oxides were white in colour. The average diameter of the particles measured by X–ray diffractometer and electron microscope were less than 100 nm except those of Al and Al_2O_3 powder. The Al_2O_3 particles were the biggest and their average size was 104 nm. The MoO_3 Particles were finest, and their average size was only 5 nm (Table 1). From this table, we can find a relationship between the particles size and the melting temperature of raw materials. The higher the temperature, the finer the particles. This is due to the low melting temperature which would make the laser atomization easier and the formation of larger particles possible. The particles size depends not only on the melting temperature , but also their heat capacity , the higher the heat capacity, the larger the particle size. Also, thermal conductivity of these materials would affect the particle size.

Table 1 The Constitution and particles size of powder prepared by irradiating laser beam

material	atmosphere	Constitution of powder	average particle size, nm
pure Al	O_2	$\eta-Al_2O_3$+Al	104
	N_2	Al	
pure M_0	O_2	$\eta-M_0O_3$	5
	N_2	M_0	
Ti6Al4V	O_2	$\alpha-TiO_2+\beta-TiO_2$	52
	N_2	Ti+TiN	50
pure Si	O_2	amorphous SiO_2+Si	56
	N_2	Si	

Phase analysis by X–ray shows that the products formed by irradiating laser bean on pure Al in 1 atm. of O_2 were 40% $\eta-Al_2O_3$, and the rest are Al particles (see Fig.1). For pure Mo, the products were 100% $\eta-MoO_3$, whose crystalline structure is still unknown (see Fig.2)[4]. For Ti6Al4V, the products consists of $\alpha-TiO_2$ and $\beta-TiO_2$, they have tetragonal structure and their lattice parameter are

$\alpha-TiO_2$: $a=0.459{,}3\pm0.000{,}2$ nm

$C=0.295{,}9\pm0.000{,}2$ nm

$\beta-TiO_2$: $a=0.378{,}5\pm0.000{,}3$ nm

$c=0.951{,}4\pm0.000{,}3$ nm

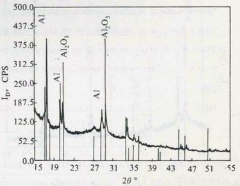

Fig.1 The X–ray diffraction diagram of Al_2O_3 particles

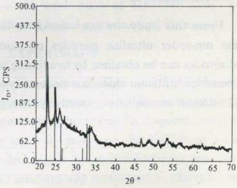

Fig.2 The X–ray diffraction diagram of M_0O_3 particle

For pure Si, the product consists of amorphous SiO_2 and a small amont of crystalline Si powder (see Fig.4). A high yield on oxides from above experiments indicates that the raw materials used in this experiments have a high O_2 affinity, so that most of them formed oxides in the nm-order, and they were crystaline states except Si.

When experiments were carried out with 1 atm. of N_2, phase analysis shows that only 70% TiN formed from Ti6Al4V and nitrides can be formed from other materials.

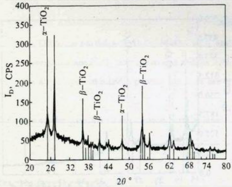

Fig.3 The X-ray diffraction diagram of TiO_2 particle

4 CONCLUSION

From this study, we can summarize that some nm-order ultrafine particles of oxides and nitrides can be obtained by irradiating laser beam on different materials under oxygen and nitrogen atmosphere respectively. A higher O_2 affinity of Mo, Si, Al and Ti make it easier to form oxides. Moreover, microcluster were found in some materials prepared by laser atomization[5], this may be a new direction for the development of new materials.

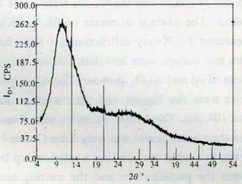

Fig.4 The X-ray diffraction diagram of SiO_2 particles

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