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Laser Production of Ultrafine Particles of Ceramics and Ceramic-Metal Mixtures†

Akira MATSUNAWA* and Seiji KATAYAMA**

Abstract

This paper describes the production and characteristics of ultrafine particles of ceramics and metal-ceramic mixtures by pulsed YAG laser evaporation technique using plates and powders of various ceramics, metal + C mixtures, metals and alloys as target materials. Most particles produced were ultrafine in the size range of less than 150 nm. The size was smaller with a reduction in chamber (ambient) pressure. It was observed that ultrafine particles were built-up to be larger due to the collision and condensation of atoms, clusters and super ultrafine particles. Ultrafine particles of β-SiC and γ-Al_2O_3 were produced from α-SiC and α-Al_2O_3 ceramic target plates in Ar atmosphere, and then the weight loss evaporated by one laser shot (evaporation rate for the formation of ultrafine particles) for ceramic targets of SiC and Al_2O_3 was by far greater than that for metal targets. Ultrafine particles of SiC and TiC could also be produced from SiC powders or Si and C mixed powders and TiC powders or Ti and C mixed powders in Ar.

Ultrafine oxide particles could be prepared from various pure metals by laser irradiation in O_2. These types were dependent upon some groups of neighboring elements: MO type of CoO, NiO, CuO and ZnO; M_2O_3 type of Cr_2O_3, Mn_2O_3, Fe_2O_3 and Al_2O_3; MO_2 type of TiO_2, ZrO_2, SnO_2 and PbO_2; M_2O_5 type of Nb_2O_5 and Ta_2O_5; MO_3 type of MoO_3 and WO_3. The formation of TiO_2 and NiO particles (of less than 10 nm in size) at 50 Pa (0.4 Torr) indicated that oxide powders could be easily formed even at a reduced pressure of up to 50 Pa. In N_2 atmosphere, ultrafine nitride particles of TiN, ZrN, VN, Cr_2N, Ta_2N, Nb_4N_3 and Mn_4N (or δ-MnN) were formed, and ultrafine particle mixtures of Al metals and AlN nitrides were made from Al plate, but metallic particles were produced from the plates of Fe, Ni, Cu, W, Mo, Si, etc. Accordingly, for Fe-Ti and Ni-Ti alloys in N_2 atmosphere, metal-nitride mixture particles such as Fe and TiN and Ni and TiN were formed, and the mixture ratio depended upon alloy composition of Fe and Ni to Ti.

KEY WORDS : (Lasers) (Ultrafine Particles) (Ceramics) (Carbides) (Oxides) (Nitrides) (Powder Production) (Evaporation Process) (Compounds)

1. Introduction

Recently, ceramics such as Al_2O_3, ZrO_2, Fe_2O_3, SnO_2, Si_3N_4, AlN, TiC, WC and SiC are called "new" or "fine" ceramics and have been drawn attention to as high performance materials because of their superior properties of high-temperature strength, high resistance to oxidation, corrosion, erosion and wear, magnetic, specific electrical and optical properties, high hardness, low density, semi-conductivity, and so on[1]. Especially, ultrafine ceramic particles of less than 1 μm in size are highlighted as high-performance materials for the achievement of excellent function parts with high reliability[2,3].

There are many physical and chemical processes for the production of ultrafine powders[2-23]. Such ultrafine powders can be produced during heating and evaporation process or absorption and reaction process by laser irradiation[4-15]. Uyeda, et al[4]. and Kato[5] attempted to produce various ultrafine oxide particles from their target oxides by cw CO_2 laser irradiation. Haggerty, et al[6,7]. tried to form ultrafine Si particles from SiH_4, and SiC or Si_3N_4 powders from SiH_4 and C_2H_4 or SiH_4 and NH_3, respectively, by CO_2 laser-assisted chemical reaction.

The authors have performed a series of fundamental studies on the formation and characterization of many kinds of ultrafine particles by laser heating and evaporation dry process[9-12]. In the previous paper[12] were clarified the formation behavior of ultrafine metal and alloy particles, laser irradiation conditions for metal particle produciton, the characteristics of shape, size distribution and crystal structure of metals, the effect of environmental pressure on the particle size.

As above-mentioned, ceramics of oxides, nitrides and carbides attract attention. Therefore, this study was undertaken to confirm a formation feasibility of ultrafine particles of various ceramics and metal-ceramic mixtures by laser evaporation technique by employing various kinds of plates and powders of ceramics, metals and alloys. The characteristics of morphology, size distribution and lattice structures of ultrafine particles were investigated with transmission electron microscope (TEM) and X-ray diffractometer.

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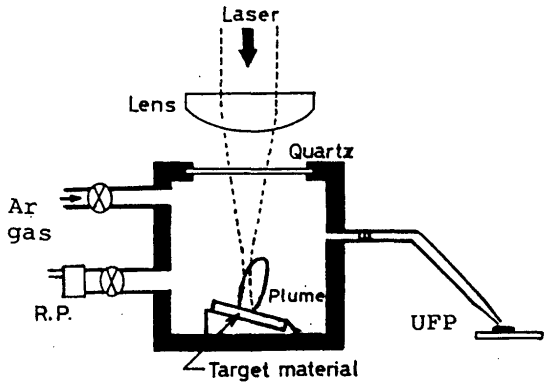


Fig. 1 Schematic illustration of laser evaporation technique.

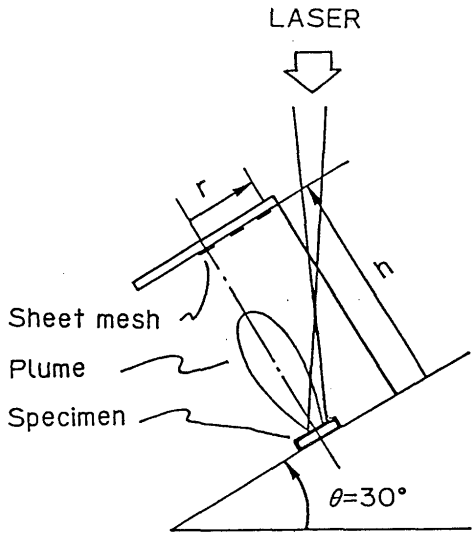


Fig. 2 Schematic arrangement of meshes for particle collection to laser-induced plume.

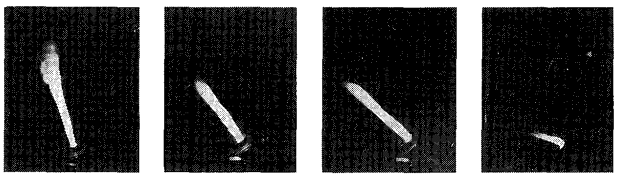


Fig. 3 Laser-induced plume from SiC target with inclinations of 10, 30, 50 and 70°.

2. Materials and Experimental Procedure

2.1 Materials used

Target materials used are α-SiC and α-Al_2O_3 (3mm^t) ceramic plates, SiC and TiC ceramic powders, Ti or Si and C metallic powders, various pure metal plates of Al, Fe, Ti, Ni, Cr, Zr, Si, Nb, Mo, Ta, W, etc., and Ni-Ti and Fe-Ti binary alloy plates (about 20, 40, 55, 65 and 80 wt%Ti for Ni-Ti alloys; 20, 33, 54, 70 and 85 wt%Ti for Fe-Ti alloys). Target plate surfaces were polished with No.400 emery paper and cleaned by acetone prior to laser irradiation.

2.2 Laser apparatus and experimental procedure

Ultrafine particles were produced by a pulse laser evaporation technique, as shown schematically in **Fig. 1**. The details of the apparatus and experimental procedure were described in the previous paper[12]. The laser used as a heat source is a pulsed Nd:YAG laser by Control Laser Model 428 (maximum average power of 200 W), which was fired at the laser pulse energy of 9 to 33 J/p, a pulse width of 3.1 ms and a repetition rate of 10 to 4 pulses/s (pps).

Moreover, in order to obtain a fundamental knowledge of the formation process of ultrafine particles (collision, condensation and coalescence of vapors), collection meshes were used, as the arrangement in the chamber is shown in **Fig. 2**.

3. Experimental Results and Discussion

3.1 Yield of ultrafine powders

Ultrafine powders were sometimes deposited and attached to the window of the chamber and its protection glass, leading to the absorption of incident laser energy and the damage of the window by the interaction of a laser beam, when the target was horizontally put parallel to the glass window in producing ultrafine particles, especially under the conditions of high laser power/energy and reduced pressure. Therefore, with the objective of improving these defects, the effect of target angle to the incident beam was first investigated.

Figures 3 (a) to (d) are still pictures showing plumes generated from the SiC target plate with inclinations of 10 to 70° by laser under the conditions of E_o = 9.2 J/P and fd = 20 mm. With each shot, a plume of intense white light emission could be observed normal to the target surface. As the angle of inclination increases, the plume was shorter, leading to lower yield of ultrafine powders with an increase in the angle of inclination. As a result, a slope of 20 to 30° was judged to be suitable for this laser production of powders.

Yields of powders were investigated by comparing weight losses evaporated by a single laser shot. **Figure 4** shows weight losses of SiC and Al_2O_3 evaporated by laser shot with E_o = 14.9 J/p in 0.1 MPa (1 atm) Ar atmosphere. Yields of particles from ceramic targets were a few times larger than for those from metal plates[12], irrespective of a small incident laser energy. It was therefore understood that it was easier to produce

powders from ceramic targets presumably because of higher laser absorption.

3.2 Production of ultrafine ceramic particles from ceramic plates and powders

3.2.1 Production from ceramic plate

Characterisitics of ultrafine particles were investigated by laser shots on Al_2O_3 and SiC plates in air, O_2 or Ar atmosphere.

Figure 5 exhibits TEM photo of particles produced from Al_2O_3 plate in Ar. Particles obtained are ultrafine in the globular shape of less than 80 nm size. **Figure 6** indicates X-ray diffractometer results of Al_2O_3 target plate (a) and ultrafine powders produced (b). γ-Al_2O_3 particles were formed although the target was α-Al_2O_3. Such Al_2O_3 particles were also produced in air and O_2.

On the other hand, SiC particles could not be prepared from SiC plate in air or O_2, because of oxidation. This suggests that inert gas atmosphere is needed to prevent oxidation. **Figure 7** shows TEM photo (a) and size

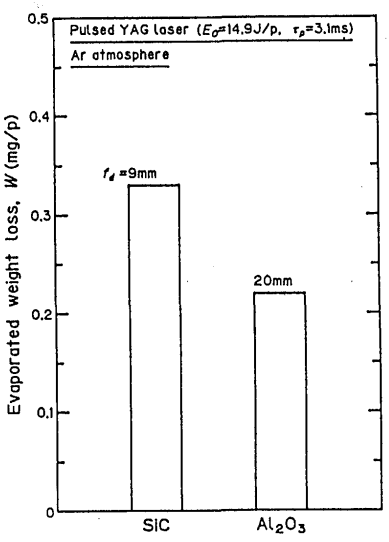


Fig. 4 Evaporation weight losses of SiC and Al_2O_3 plate targets by laser shot in Ar.

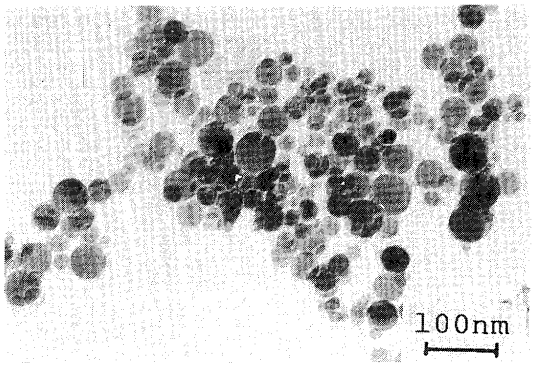
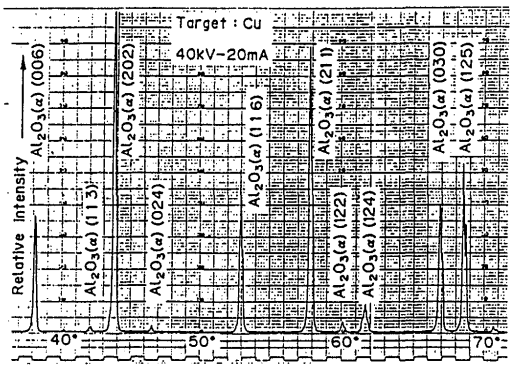
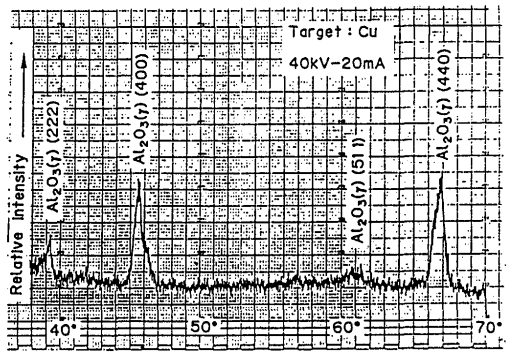


Fig. 5 TEM photo of particles produced from Al_2O_3 plate target by laser in Ar.

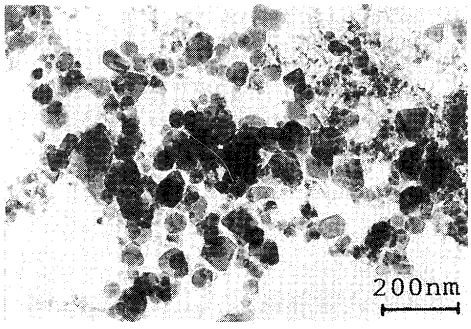


(a)

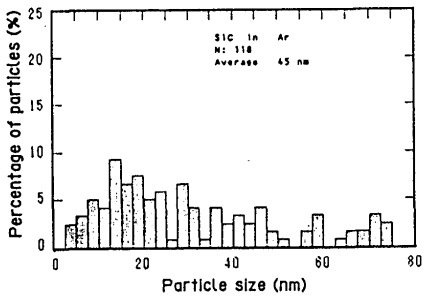


(b)

Fig. 6 X-ray diffractometer results of Al_2O_3 plate target (a) and ultrafine powders produced (b).

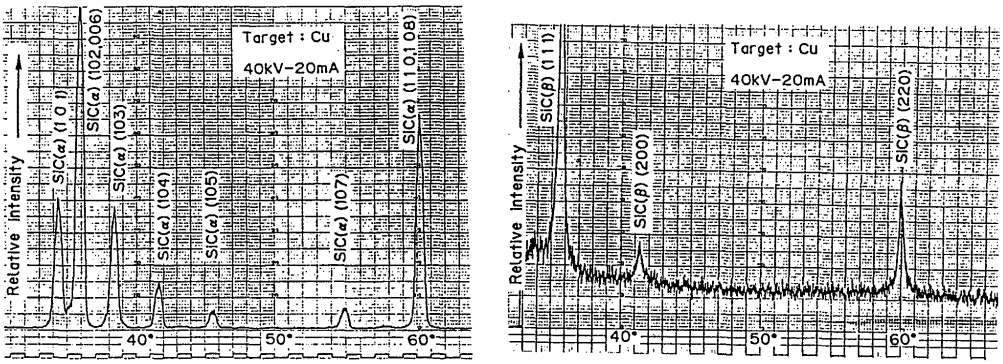


(a)



(b)

Fig. 7 TEM photo (a) and particle size distribution (b) of powders made from SiC target by laser in Ar.



(a) (b)

Fig. 8 X-ray diffractometer results of SiC plate target (a) and ultrafine powders produced (b).

distribution (b) of particles made from SiC plate in Ar. **Figure 8** indicates X-ray diffractometer results of SiC target plate (a) and ultrafine powders prepared from it (b). Granular, polygonal β-SiC particles in a wider size range of less than 150 μm were formed from the target of α-SiC.

3.2.2 Production from ceramic powders

Ultrafine particles were tried to produce from powders in Ar. X-ray diffraction result of ultrafine particles prepared from SiC powders is shown in **Fig. 9**. It is noted that a small number of Si particles were formed in addition to β-SiC ones. This suggests that most or part of SiC target powders are decomposed into Si and C atoms during evaporation, and that Si and C elements are not completely united or combined again. The extra amount of C is required to avoid the formation of Si particles and to form more ultrafine SiC particles. In fact, the formation of Si particles was prevented by using powders mixed at the weight ratio of SiC: C = 20: 1. This result suggests a feasibility of SiC particle formation from the mixture of Si and C powders. Additionally, the reason

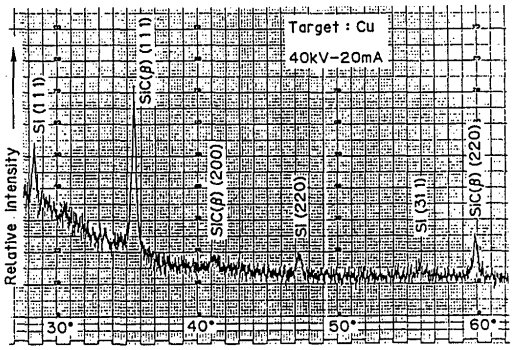
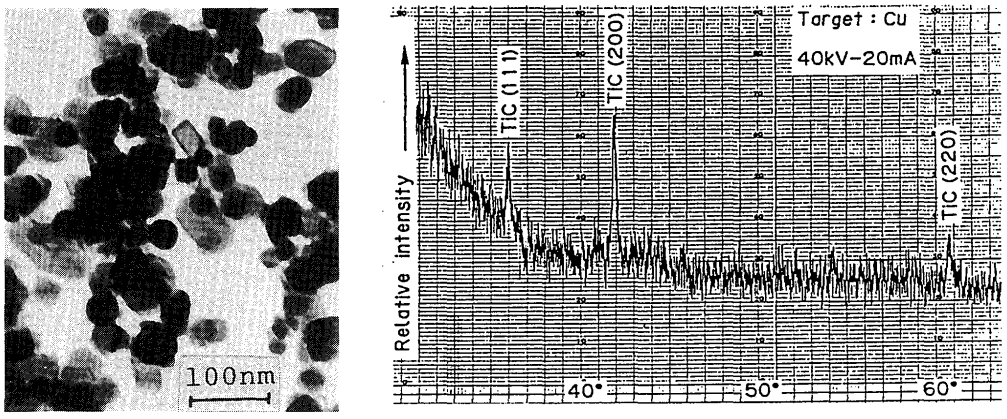


Fig. 9 X-ray diffractometer result of ultrafine particles produced from SiC powder target by laser in Ar.

why Si particles were not formed from SiC plate may be interpreted in terms of the fact that extra free C is added as a synthesis-assisting material.

Ultrafine TiC particles were produced by laser irradiation on TiC powders. Polygonal particles of the same crystal structure as the target powders were formed, as shown in **Fig. 10**. For TiC target powders, decomposed



(a) (b)

Fig. 10 TEM photo (a) and X-ray diffractometer result (b) of ultrafine TiC particles produced from TiC powder target by laser in Ar.

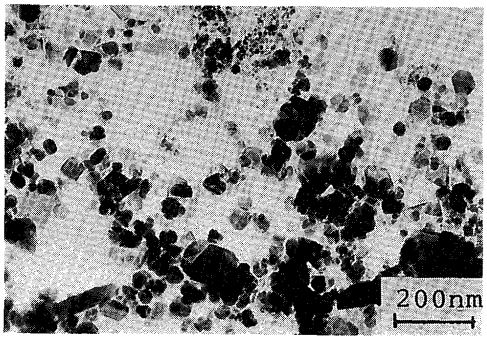
and isolated Ti was not detected, whereas decomposed Si was present for the use of SiC powders. This may suggest that the potentiality of TiC particle formation is greater than that of SiC.

3.2.3 Production of carbide particles from metal-carbon mixture powders

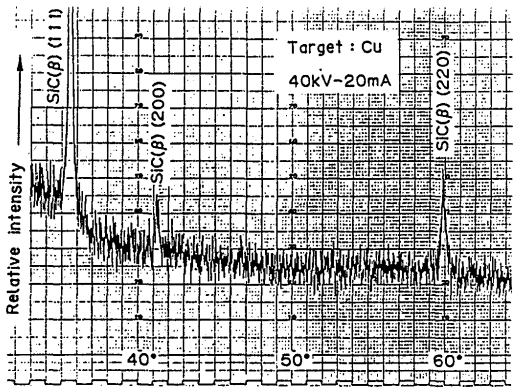
An attempt was made to produce ultrafine particles of SiC and TiC ceramics from mixtures of Si/C and Ti/C powders, respectively, by laser evaporation method in Ar atmosphere. **Figures 11** (a) and (b) show TEM photo and X-ray diffractometer result of particles prepared from the mixtures of Si/C powders at 1:1 ratio. Ultrafine particles in (a) closely resemble the particles produced from SiC plate (See Fig.7) and powders, and the formation of SiC ceramic particles is confirmed in (b). (Besides, extremely ultrafine particles observed may be of C although it can not be detected by X-ray diffractometer, and thus other analytical methods should also be utilized to sufficiently identify particles.)

It was also confirmed that ultrafine TiC particles could be produced by irradiating a laser onto mixtures of Ti/C powders in Ar. Moreover, intense exothermic reaction took place (like selfburning) in the target powders of Ti and C, leading to the spontaneous formation of TiC ceramic powders of the order of mm, although such burning was not observed in Si and C powders. It is noted that such phenomenon is used for exothermic spontaneous synthesis of ceramics, and that a laser may be useful as an ignition source.

From the above results it was found that SiC and TiC ceramic particles could be formed by using Si + C and Ti + C powder mixtures.



(a)



(b)

Fig. 11 TEM photo (a) and X-ray diffractometer result (b) of particles produced from mixture target of Si and C powders by laser in Ar.

3.3 Production of ultrafine oxide particles from metal by laser shot in oxygen

An investigation was performed to produce ultrafine oxide particles by the laser evaporation method of metal target plates in O_2 atmosphere at 0.1 - 0.2 MPa (1 -2 atm).

Figure 12 shows TEM photo (a), electron diffraction rings (b), size distribution (c) and X-ray diffractometer result (d) of particles prepared from Al plate by the laser process in O_2. Particles were identified to consist of γ-Al_2O_3 oxides mainly and δ-Al_2O_3 oxides partly without metallic phase of Al. Almost all particles were spherical in shape, and their sizes were predominantly in the range of less than 100 nm. Observation and analytical results of particles produced from Fe target plate in O_2 are given in **Fig. 13**. Particles of γ-Fe_2O_3 and ε-Fe_2O_3 oxides were formed. Most particles were spherical in the size range of 10-80 nm, and part of larger particles were polygonal in the range of 80-130 nm.

The production of various oxide particles was conducted by utilizing other metals in a similar way. The result of kinds and crystal structures of particles produced are summarized in the periodic table of elements of **Table 1**. Five types of oxide particles were produced. It is interesting to know that neighboring elements are grouped to form the same type: MO type of CoO, NiO, CuO and ZnO; M_2O_3 type of Cr_2O_3, Mn_2O_3, Fe_2O_3 and Al_2O_3; MO_2 type of TiO_2, ZrO_2, SnO_2 and PbO_2; M_2O_5 type of Nb_2O_5 and Ta_2O_5; MO_3 type of MoO_3 and WO_3, where M is representative of metallic element. As well known, Al_2O_3, Fe_2O_3, TiO_2, ZrO_2, etc. have various kinds and crystal structures named {α, β, γ, δ, ε, etc.}, {rutile, anatase, brookite} and {monoclinic, tetragonal, cubic}. It is understood that, in laser evaporation process, phases stable at high temperature might be formed and retained as metastable at room temperature.

Oxide particles were slightly larger in size and had broader size distribution than metal ones. It is interesting to know whether oxide particles are formed from metal target in O_2 at a low pressure or not. **Figure 14** exhibits TEM photo of ultrafine particles made from Ti in O_2 at 1.3 kPa (10 Torr). X-ray diffractometer results of particles produced at 0.1 MPa (760 Torr), 1.3 kPa (10 Torr) and

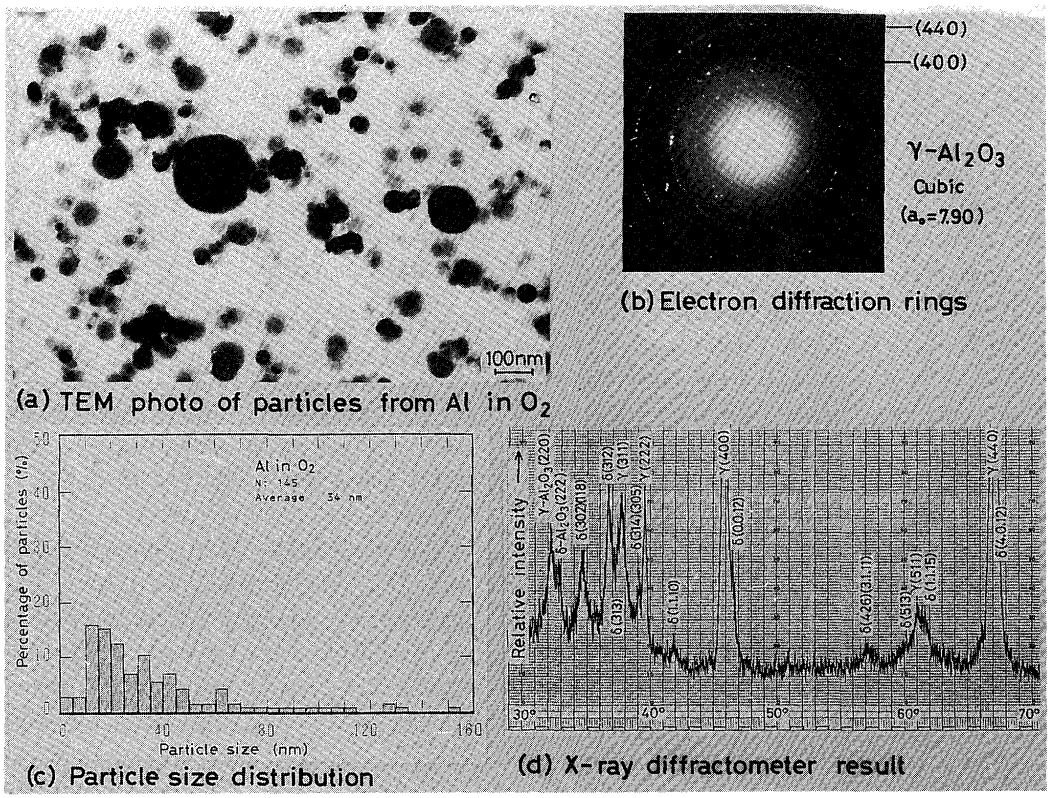


Fig. 12 TEM photo (a), electron diffraction rings (b), particle size distribution (c) and X-ray diffractometer result (d) of powders produced from Al plate target by laser in O_2.

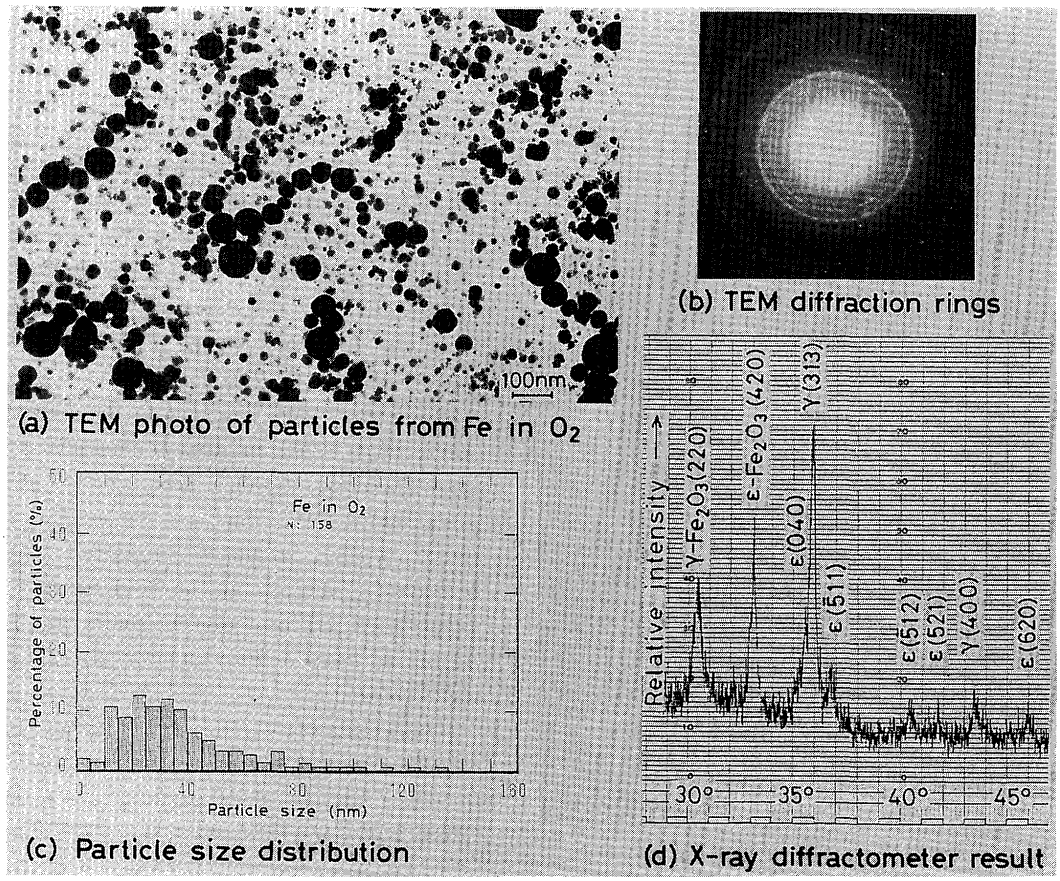


Fig. 13 TEM photo (a), electron diffraction rings (b), particle size distribution (c) and X-ray diffractometer result (d) of powders produced from Fe plate targer by laser in O_2.

Table 1 Summary of ultrafine oxide particles produced by laser evaporation method of various metals in O_2 tabulated in periodic table of elements

	IVB	VB	VIB	VIIB	VIII	VIII	VIII	IB	IIB	IIIA	IVA
3										Al	Si
										Al_2O_3 *1	—
										(Cubic)	[Amor]*2
4	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn		Ge
	TiO_2 *3	—	Cr_2O_3	γ-Mn_2O_3	Fe_2O_3 *4	CoO	NiO	CuO	ZnO		—
	(Tetra)	[Amor]*2	α-Al_2O_3	(Tetra)	(Tetra)	NaCl (C)	(Hexa)	(Mono)	ZnO (H)		[Amor]*2
5	Zr	Nb	Mo								Sn
	ZrO_2 *5	Nb_2O_5	η-MoO_3								SnO_2
	(Mono)	(Mono)	(-)								(Tetra)
6		Ta	W			Element					Pb
		δ-Ta_2O_5	WO_3			UFP oxides produced					PbO_2 *6
		(Ps.H)	(Tricl)			Crystal structure (system*7)					(Orthor)

Note : #1 γ-Al_2O_3(C)+δ-Al_2O_3(-) #2 Amorphous #3 Anatase(Tetra)+Rutile(Tetra)
#4 γ-Fe_2O_3(Tetra)+ε-Fe_2O_3(Mono) #5 Cubic+Tetragonal+Monoclinic #6 α-PbO_2(+PbO(Orthorhombic))
#7 C:Cubic; Tetra:Tetragonal; H & Hexa:Hexagonal; Mono:Monoclinic; Tricl:Triclinic; Orthor:Orthorhombic; Ps.H:Pseudo Hexagonal

0.1 kPa (1 Torr) are shown in **Fig. 15**. (Broad peak of 20-40° is due to the glass for powder collection.) It was clearly found that TiO_2 oxide particles (of less than 10 nm in size) could be produced in O_2 even at 0.1 kPa. Also, the production ratio of rutile to anatase TiO_2 increased with a decrease in pressure. This may presumably have a correlation to particle size and quenching rate from high temperature. Similarly, it was confirmed that TiO_2 and NiO could be formed from Ti and Ni targets in O_2 at a reduced pressure of up to 50 Pa (0.4 Torr: minimum low pressure at this experiment).

3.4 Production of ultrafine nitride particles from metal by laser shot in nitrogen

A feasibility of the formation of ultrafine nitride particles was investigated by laser evaporation of metal targets in N_2 atmosphere at 0.1 MPa (1 atm).

Figure 16 shows TEM Photo and X-ray diffractometer result of ultrafine particles produced from Cr plate in N_2.

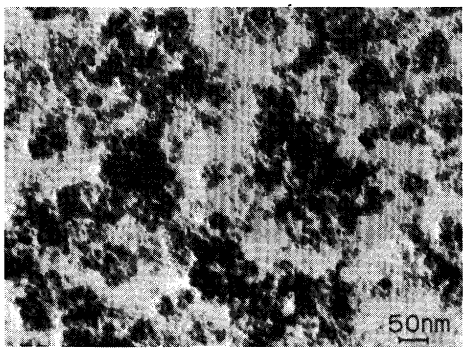


Fig. 14 TEM photo of ultrafine powders produced from Ti plate target by laser in O_2 at 1.3 kPa (10 Torr).

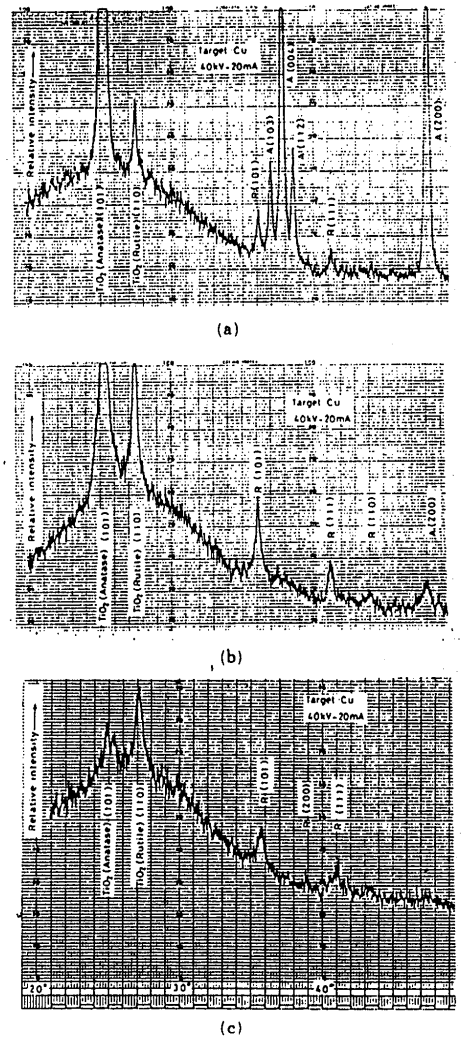


Fig. 15 X-ray diffractometer results of ultrafine powders produced from Ti plate target by laser in O_2 at 0.1 MPa (a), 1.3 kPa (b) and 0.1 kPa (c).

Spherical β-Cr_2N ceramic particles of less than 100 nm in size were formed. **Figure 17** shows TEM photo (a), size distribution (b) and X-ray diffractometer result (d) of particles produced from Al plate. Two characteristic types of smaller polygonal particles with edges and slightly larger spherical particles were observed, in accordance with two ranges of 10-60 nm and 60-120 nm in particle size distribution. These may correspond to AlN ceramic particles and Al metallic particles respectively.

Such similar investigation was undertaken using other metal targets in N_2 atmosphere. Summary results of kinds and crystal structures of particles produced by laser evaporation technique in N_2 atomosphere are given in part of periodic table of elements in **Table 2**. For Ti, Zr,

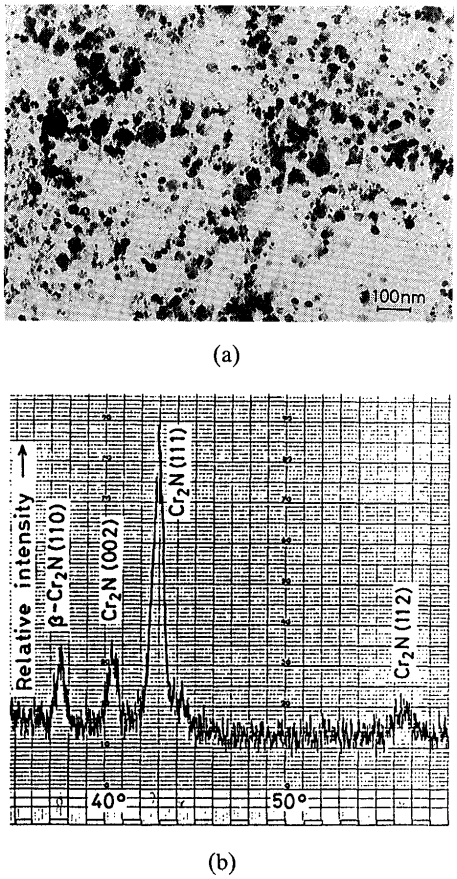


Fig. 16 TEM photo (a) and and X-ray diffractometer result (b) of powders produced from Cr plate target by laser in N_2.

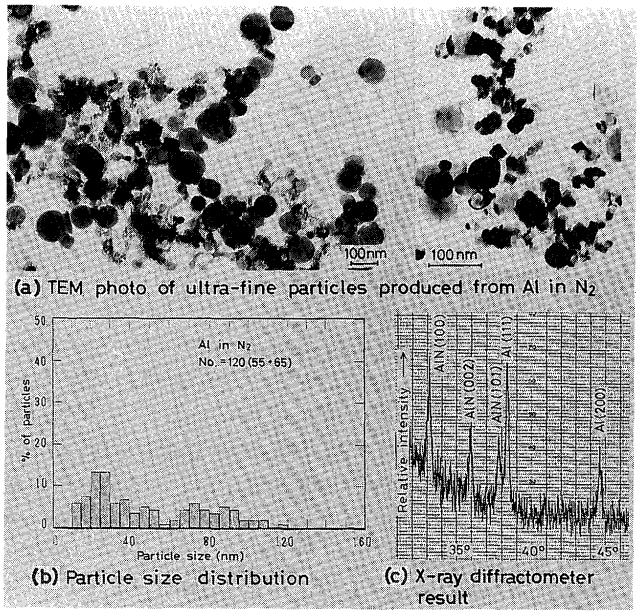


Fig. 17 TEM photo (a), particle size distribution (b) and X-ray diffractometer result (c) of powders produced from Al plate target by laser in N_2.

Table 2 Ultrafine nitride or metal particles produced by laser evaporation process of various metals in N_2 summarized in periodic table of elements

	IVB	VB	VIB	VIIB	VIII	VIII	VIII	IB	IIB	IIIA	IVA
3										Al	Si
										Al+AlN*1	Si
	IVB	VB	VIB	VIIB	VIII	VIII	VIII	IB	IIB	(FCC)(H)	(Diamo)
4	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn		Ge
	TiN	VN	β-Cr2N	Mn4N *2	α-Fe	Co(CoO)	Ni	Cu	Zn		Ge
	NaCl(C)	NaCl(C)	(Hexa)	(Cubic)	(BCC)	(FCC)(C)	(FCC)	(FCC)	(HCP)		(Diamo)
5	Zr	Nb	Mo								Sn
	ZrN	Nb4N3 *3	Mo								β-Sn
	NaCl(C)	(Tetra)	(BCC)								(Tetra)
6		Ta	W			Element					Pb
		Ta2N	W,(W3O)			UFP produced in N2					Pb
		(Hexa)	(BCC) *4			Crystal structure (system*5)					(FCC)

Note : #1 Al(FCC)+AlN(ZnO struc.(Hexa)) #2 Mn4N(Cubic)+δ-MnN(Tetra) #3 Nb4N3(Tetra)+Nb4m3.82(Cubic)
#4 W(BCC)+W3O(β-W)
#5 C:Cubicic; Tetra:Tetragonal; H & Hexa:Hexagonal; Diamo:Diamond.

V, Nb, Ta, Cr and Mn targets, nitride particles of TiN, ZrN, VN, Nb_4N_3, Ta_2N, Cr_2N and Mn_4N (or δ -MnN) were formed, respectively. Ultrafine particle mixtures of Al metals and AlN nitrides were made from Al. However, for Fe, Co, Ni, Cu, Zn, W, Mo, Si, Ge, Sn and Pb elements, the powders produced were not nitrided but metallic, and they were the same particles as in Ar. There appears to be a tendency that MN(M:metallic element) type was formed for Ti and its vicinity in the periodic table, and the ratio of N to M decreased just like M_2N type for elements far from Ti and M type for elements farther away.

Free energies for formation of several nitrides are shown as a function of temperature in **Fig. 18**[24]. This indicates that TiN and ZrN were likely to form due to great negative free energy. It is also understood that Cr_2N, which is more stable above 600 K than CrN, was produced, and that it was difficult to produce Fe-nitrides such as Fe_2N and Fe_4N because of thermodynamical instability above room temperature. These results suggest that this laser evaporation technique in N_2 may produce mixtures or composites of ultrafine metal + nitride powders by using an alloy target consisting of a metal-forming element and a nitride-forming element.

3.5 Production of ultrafine metal + nitride particle mixtures from alloy by laser shot in nitrogen

A formation feasibility of mixtures or composites of ultrafine metal-nitride particles was investigated by laser evaporation of Ni-Ti and Fe-Ti alloys in N_2. **Figure 19** indicates X-ray diffractometer results of powders obtained from 80%Fe-20%Ti (FT2), 46%Fe-54%Ti (FT5) and 15%Fe-85%Ti (FT8) alloys. Fe and TiN phases were formed from Fe-Ti alloys, and the peak ratio of TiN nitride to Fe metal varied with alloy composition of Ti to Fe. Similar results were also obtained from Ni-Ti alloys. **Figure 20** (a) and (b) exhibit TEM photos of particles produced from 35%Ni-65%Ti alloy (NT6) and 46%Fe-54Ti alloy (FT5), respectively. Ultrafine particles of less than 100 nm size are observed in the projection form of cubic, polygon and globule. It is judged from particle shapes that cubic particles are regarded as TiN nitrides and spherical particles were metallic. From these results, it was found that mixtures of Fe and TiN and Ni and TiN were formed, and that the mixture ratio was controllable depending on alloy composition of a target.

3.6 Formation process of ultrafine particles

The formation process of ultrafine particles was examined by using collection Cu meshes coated with carbon film as shown in Fig. 2, when laser pulses were shot at E_o = 14.9 J/P and fd = 20 mm at 100 or 0.6 kPa. **Figure 21** exhibits TEM photos of ultrafine SiC particles collected directly by carbon films at various locations (distance from target surface to mesh, h = 7 & 45 mm, distance from plume axis to mesh, r = 0 & 5 mm) at

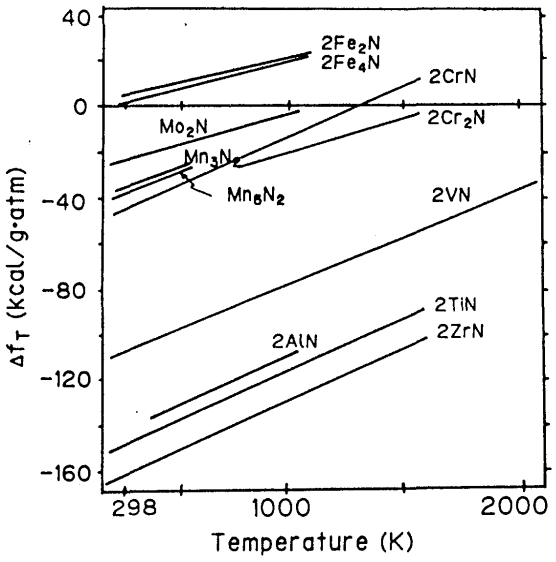


Fig. 18 Free energy of formation of several nitrides as function of temperature[24].

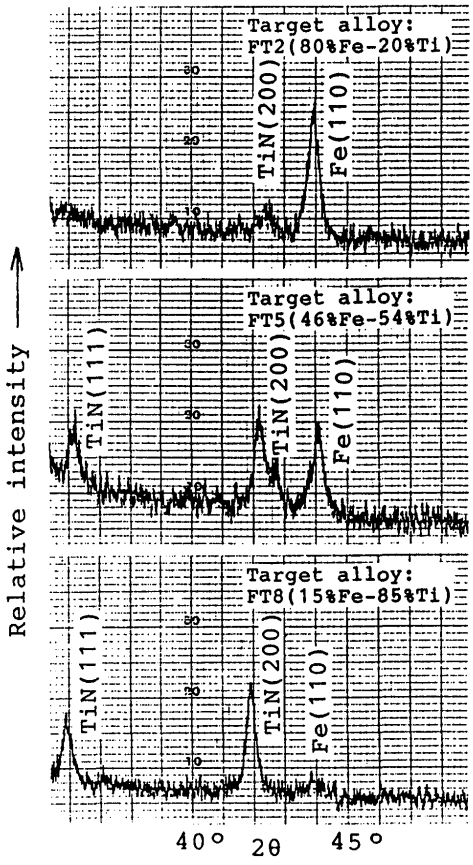


Fig. 19 X-ray diffractometer results of powders produced from targets of 80%Fe-20%Ti, 46%Fe-54%Ti and 15%-85%Ti alloys by laser in N_2.

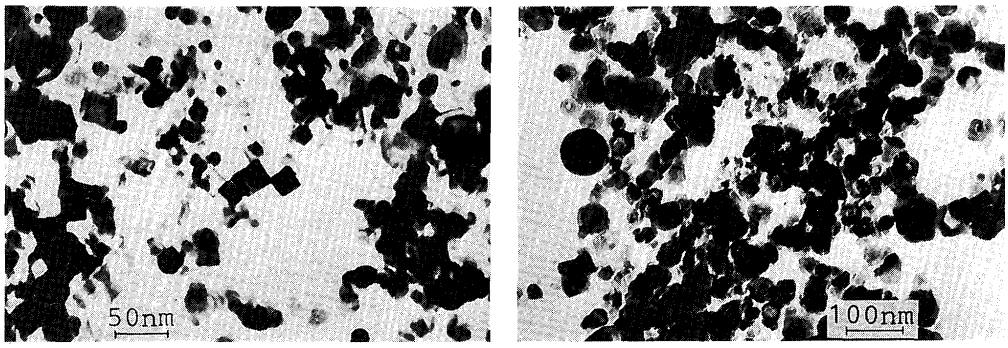


Fig. 20 TEM photos of ultrafine powders produced from targets of 35%Ni-65%Ti and 46%Fe-54%Ti alloys by laser in N_2.

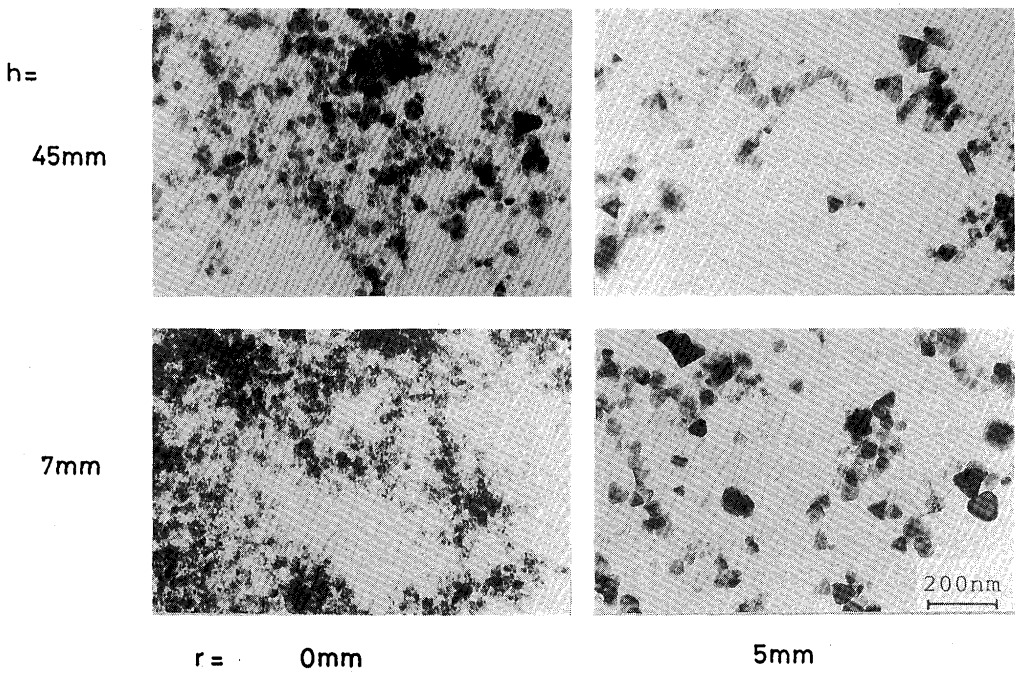


Fig. 21 TEM photos of ultrafine SiC powders produced from SiC plate target by laser in Ar at 100 kPa, and collected directly by carbon film on mesh at locations of h = 7 & 45 mm and r = 0 & 5 mm.

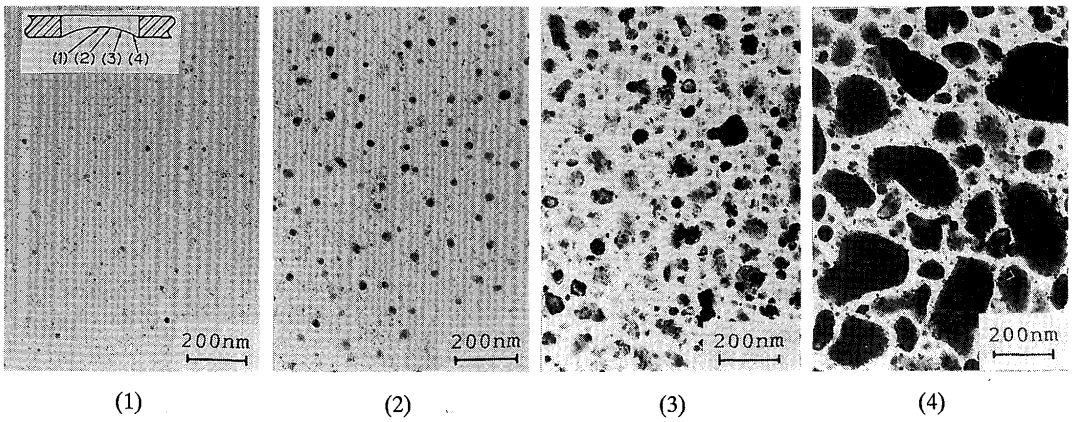


Fig. 22 TEM photos of ultrafine Ni powders produced from Ni plate target by laser in Ar at 0.6 kPa and collected directly by concave carbon film in mash hole.

100kPa. Larger particles were observed in the outer part of a plume. **Figure 22** shows TEM photos of Ni particles produced at 0.6 kPa, collected through concave carbon film and observed from center to outer in one mesh hole (h = 9 mm, r = 0 mm). Particles were larger away from the center of the mesh hole. This suggests that the collision and condensation (coalescence) of vapors took place during flow on (below) the concave film (during flying upwards from the target).

4. Conclusions

A production feasibility of ultrafine particles of ceramics and metal-ceramic mixtures was investigaed by pulsed YAG laser heating-evaporation technique of various target materials in Ar, O_2 or N_2 atmosphere. The following chief conclusions can be drawn:

(1) Ultrafine particles of β-SiC and γ-Al_2O_3 ceramics could be produced from their α-SiC and α-Al_2O_3 ceramic plates in Ar atomosphere.

(2) The weight losses of SiC and Al_2O_3 ceramic targets evaporated by one laser shot (corresponding to evaporation rate for the formation of ultrafine particles) were by far greater than those of metal targets.

(3) Ultrafine particles of SiC and TiC could be produced from SiC powders or Si and C mixed powders and TiC powders or Ti and C mixed powders in Ar.

(4) Ultrafine oxide particles could be formed from various pure metals by laser irradiation in O_2.

(5) Oxides with 5 types of metal-to-oxygen ratios appeared to form according to neighboring similar elements: MO type of CoO, NiO, CuO and ZnO; M_2O_3 type of Cr_2O_3, Mn_2O_3, Fe_2O_3 and Al_2O_3; MO_2 type of TiO_2, ZrO_2, SnO_2 and PbO_2; M_2O_5 type of Nb_2O_5 and Ta_2O_5; MO_3 type of MoO_3 and WO_3.

(6) TiO_2 and NiO oxide particles (of less than 10 nm size) were produced from metal in O_2 even at reduced pressure of 50 Pa (0.4 Torr).

(7) Almost all ultrafine particles were in the size range of less than 150 nm. The size was smaller with a reduction in chamber (ambient) pressure.

(8) In N_2 atmosphere, ultrafine nitride particles of TiN, ZrN, VN, Cr_2N, Ta_2N, Nb_4N_3 and Mn_4N (or δ-MnN) were produced from their metal targets, and ultrafine particles mixtures of Al metals and AlN nitrides were made from Al plate, but metalic particles were produced from the plates of Fe, Ni, Cu, W, Mo, Si, etc. This result was interpreted in terms of thermodynamic stability (free energy for formation).

(9) For Fe-Ti and Ni-Ti alloys in N_2 atmosphere, metal-nitride mixture particles such as Fe and TiN and Ni and TiN were formed, and the mixture ratio depended upon alloy composition of Fe and Ni to Ti.

(10) The formation process of ultrafine powders was understood in terms of collision, condensation and coalescence of vapors.

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