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Spectroscopic Measurement of Plume in Laser PVD Process†

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In recent years, so called CVD (Chemical Vapor Deposition) and PVD (Physical Vapor Deposition) methods are spotlighted as hopeful techniques for surface modification in producing ceramic films on the surface of metallic and/or non-metallic materials.

In comparison with CVD method, PVD method has such advantages that 1) higher deposition rate can be obtained, 2) lower substrate preheating temperature which does not spoil the characteristics of the substrate material is acceptable and 3) poisonous reactive gases which yield ceramic vapor are not required¹⁾.

From the reason that laser beam is an ideal energy source in that it can yield such a high energy density as to evaporate ceramic materials easily, it has been proposed to utilize a high power laser for PVD of ceramics²⁾.

In ordinary laser PVD method, however, there is a problem that the structure and/or composition of the deposited film is different from that of the ceramics used as a target^{2),3)}. For example, when BN ceramics is used as the target, the percentage of nitrogen in the deposited film is decreased compared with that in the target. In order to solve the problem, the technique which supply N⁻ ion into a vacuum chamber through an ionizing-gun during the vaporization of BN target has been tried²⁾. It has been shown that the technique works satisfactorily to produce ceramic film which is stoichiometrically the same composition with BN used as the target.

It is important to know the vaporization phenomenon of ceramics in order to understand above-mentioned result and to improve the characteristics of the deposited ceramic films.

The object of this study is to clarify the vaporization phenomena of ceramics by spectroscopic measurements of brilliant plumes which are induced by laser irradiation. Continuous wave CO₂ and Q-switched YAG lasers are

used in the experiment and the effect of laser condition on the vaporization phenomena is discussed.

Si₃N₄ ceramics were used as the target material. Continuous wave CO₂ laser was operated at the output power of 80W, and Q-switched YAG laser was mainly used at 12W average output with high peak power. For reference, Q-switched YAG laser was also used at 80W average output with lower peak power. Spectroscopic measurement was conducted using a monochromator available for the wave length between 300 and 900nm. The pressure in the chamber was about 100Pa.

Figures 1(a) and (b) show the still pictures of induced plumes by CO₂ laser and high peak power Q-switched YAG laser, respectively. Detrimental spattering can not be observed at each focal point shown in the figure. Although colors of the plumes can not be seen from the monochrome photos, they both are light violet. However, the reflection light colors from the surroundings such as the jig and lens holder which are close to laser irradiating spots are quite different each other. That is, the reflection light color of the plume shown in Fig. 1(a) is orange while it is light violet in (b). The difference of the color makes us imagine that there is something different in the vaporization phenomenon between the plumes.

Figures 2(a) and (b) show the results of spectroscopic measurement of plumes induced by CO₂ and high peak power Q-switched YAG lasers, respectively. Although several spectrum lines related to oxygen can be observed in the figure, we discuss the states of silicon and nitrogen which are the constituent elements of Si₃N₄ ceramics hereafter. In the wave length range of 380 ~ 440nm spectrum lines of NI, NII, NIII, SiI, SiII, SiIII and SiIV can be expected to be found if they are in the plume. Supplemental marks I, II, III and IV represent the states of

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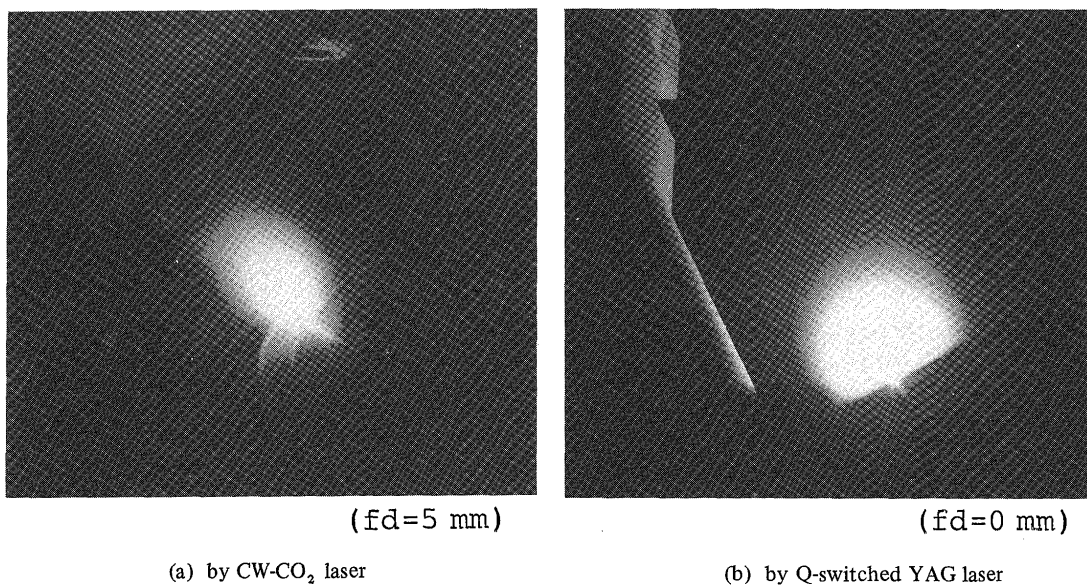


Fig. 1 Still pictures of plume induced by laser irradiation on Si₃N₄

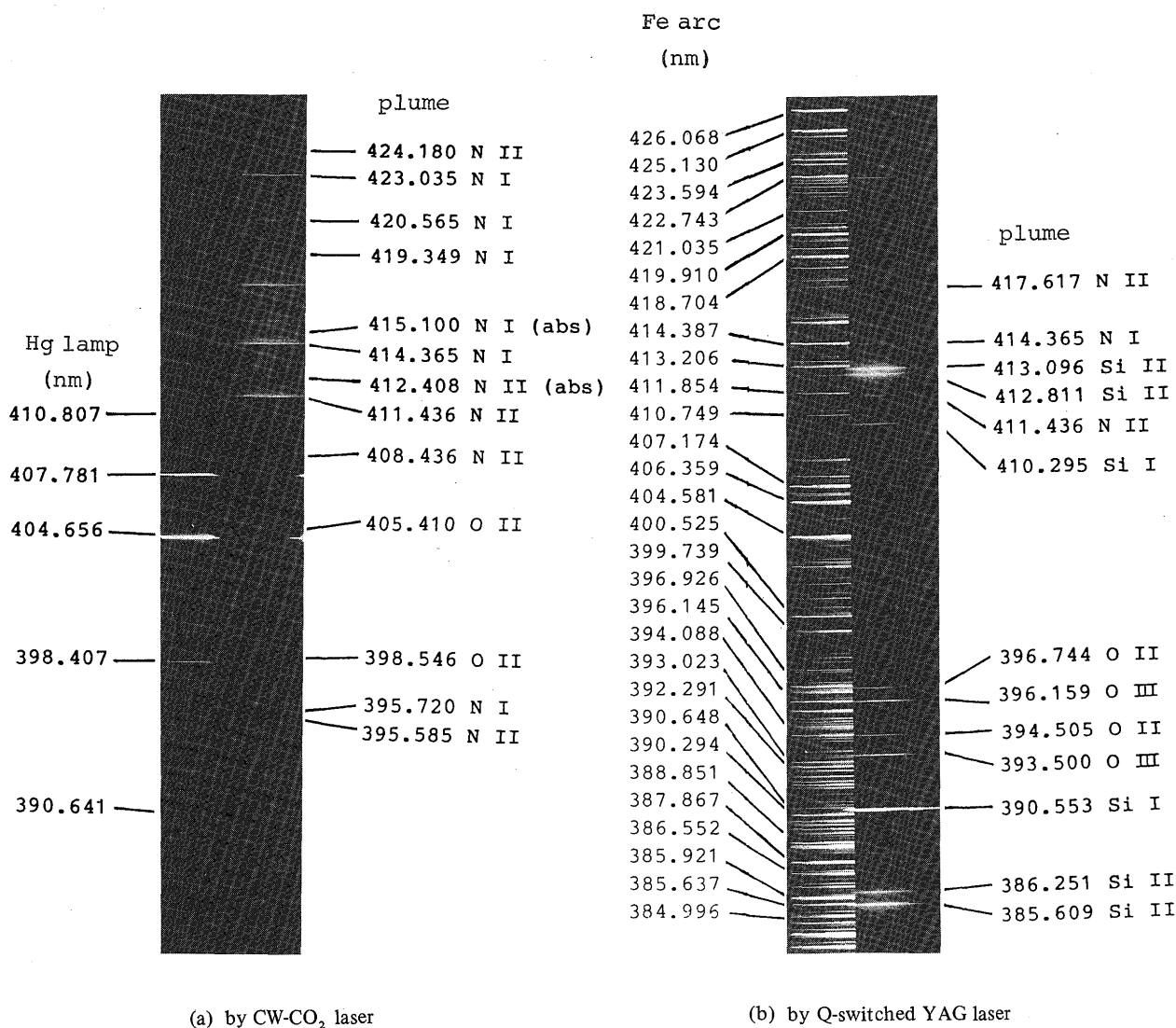


Fig. 2 Spectrum lines of plume induced by laser irradiation on Si₃N₄

ionization of each element. That is, I is used for normal atom, and II, III and IV for single, second and third state of ionization, respectively⁴⁾. There are two rows of spectrum lines in each photo in Fig. 2. Spectrum lines of Hg lamp and Fe arc are used as reference lines in (a) and (b), respectively.

In Fig. 2(a), spectrum lines of NI and NII and a band spectrum which is considered to be emitted from molecular nitrogen can be observed. Absorptive lines of wave lengths 415.100 and 412.408 nm which deserve NI and NII, respectively, are also observed. Accordingly, it is considered that both excitation and absorption phenomena of NI and NII are happening in the molecular nitrogen atmosphere induced by the pyrolyzation of Si_3N_4 . On the other hand, none of Si lines can be observed here. Si seems to be in the ground state in this case.

In Fig. 2(b), spectrum lines of SiI and SiII (which can not be found in (a)) are observed together with the lines of NI and NII. Band spectrum can not be observed here. in the case of high peak power Q-switched YAG laser,

it was found that there existed both ionized Si and N in the plume.

Although not shown in the figure, the distribution of spectrum lines obtained from the plume induced by lower peak power Q-switched YAG laser is the same with that by CO_2 laser in Fig. 2(a).

Through a spectroscopic measurement of plumes, it was confirmed that the vaporization phenomenon of Si_3N_4 in laser PVD process differed under different laser irradiation conditions. It suggests that the characteristics of the deposited film may be dependent on laser conditions. It is under investigation and discussion by the authors.

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