Spraying of MgO Films by Using a Well-controlled Plasma Jet

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Abstract

With the use of our original thermal plasma reactor, spraying of MgO films is studied. Under some experimental conditions, MgO films are well prepared although spraying of MgO is usually said to be difficult due to its particular properties. The relationship between spraying conditions including basic parameters of plasma jet (i.e. chamber pressure, jet power, jet temperature, jet velocity and spraying distance) and prepared films are briefly discussed.

Key words: plasma jet, thermal plasma reactor, plasma spraying, MgO coating

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1. Introduction

Thermal plasma processing using a plasma jet with high speed and high heat capacity under various operatin conditions is a valuable method for spraying refractory materials and synthesizing new materials [1-3]. To this end, we have newly developed a thermal plasma reactor [4, 5] composed of a forced constricted type (FC-type) plasma jet generator with a feed ring. This reactor can generate stable plasma jets with high heat capacity under various operating conditions. By using this reactor, we have performed production of ultra fine particles [4, 5], synthesis of new ceramics [1-3], and synthesis of diamond from gaseous materials [6].

Sintered MgO film is used as a substrate for a bismuth-based high temperature superconducting materials. In order to realize devices with using high temperature superconductors, coating of dense MgO films on a metallic substrate is needed [7]. But, spraying of MgO is generally difficult, and there are no reports on success of spraying MgO using a gas-stabilized plasma jet except a water-stabilized arc [7,8]. MgO has particular properties; high melting point (2800 ℃), high latent heat of melting (1920 J/cm^3), and relatively high thermal conductivity. The main difficulty is therefore heating up and melting uniformly all injected MgO particles —especially of a wider size cut - during a very short dwell time in the hot plasma flow and plasma jet [7, 8]. To realize spraying of MgO, precise controls of both plasma jet and interaction between plasma flow and injected particles are required.

In this work, we have tried spraying of MgO by using the reactor with the FC-type plasma jet generator [1, 4, 5]. The goal of this work is to test spraying MgO, to optimize the spraying conditions, and to check the structure of the prepared films. MgO coatings have been successfully prepared at certain experimental conditions. We present some related experimental results and briefly discuss the relationship between prepared MgO films and spraying conditions.

2. Experimental set up and method

A schematic drawing of the plasma jet reactor system [4, 5] used in this work is shown in Fig.1. The reactor consists of the forced constricted type (FC-type) plasma jet generator, the feed ring (FR), the reaction chamber and the powder feeder. The FC-type plasma jet generator consists of the nozzle anode (made of copper) with 5 mm diameter, the rod cathode (made of 2 % thorium-tungsten) with 5 mm diameter and the insulated constrictor nozzle (i.e. the floating electrode, made of copper) with 5 mm diameter.

As the insulated constrictor nozzle peculiar to this generator always fixes the arc length and the nozzle wall strongly constricts the arc column, the stable plasma jet with high heat capacity is produced under various experimental conditions [1, 3-5]. Experiments are performed under continuous pumping and flowing of argon gas. The plasma jet is produced by DC arc discharge. MgO powders are injected into the hign temperature region of plasma flow with carrier (feed) gas through the FR. By changing FR length, interaction length or time of injected powders with plasma flow is controlled. MgO films are prepared on the molybdenum substrate with 15 mm diameter. These films are characterized by an X-ray diffractometer and scanning electron microscope (SEM).

Experiments have been performed under the following conditions: The pressure in the reaction chamber P_t is 100 - 760 Torr; the plasma gas (Ar) flow rate Q is 20 *l*/min; the feed gas (Ar) flow rate Q_f is 4-10 *l*/min; MgO powder feeding rate Q_m is 0.5 g/min; jet power W_j is varied from 3 to 5 kW; the distance from the FR exit to substrate, namely substrate position L_s is varied from 40 to 100 mm, and spraying duration time t_s is 4 or 5 min.

3. Experimental results and discussion

Spraying of MgO films strongly depends on operating conditions, i.e. P_t, W_j, L_s, Q_f and Q_m.

At first, we want to confirm that MgO films are successfully prepared by using our well-controlled plasma jet. Experimental conditions and others are summarized in Table 1.

Phase composition of prepared films was checked by the X-ray diffraction analysis. Figure 2 shows a X-ray diffraction (XRD) patterns of raw powder materials MgO (a) and a prepared film under P_t = 760 Torr. Peak spectra of the prepared film coincide with that of MgO in (a). Some peaks of molybdenum (substrate material) are also observed. Because, during spraying, circumference of the substitute is masked by the substitute holder and this part is not sprayed. At any rate, it is confirmed that MgO coating is prepared.

Figure 3 shows XRD patterns of the prepared films as a function of L_s, where P_t = 760 Torr and W_j = 4 kW. Thickness of MgO film varies with L_s. In the downstream region (i.e. L_s = 100 mm), thickness of MgO film is too thin, and large peaks of a molybdenum substrate appear. Corresponding to the variations of XRD patterns, deposition rate increases with decreasing L_s until L_s = 60 mm. With decreasing L_s further (i.e. at L_s = 40 mm), however, thickness decreases. At about L_s = 60 mm, film thickness reaches the maximum under this conditions. Similar tendency is observed at W_j = 3 kW. Judging from these results, in the present experiment, MgO films are well prepared with L_s from 40 mm to 60mm.

To study the influence of W_j on spraying of MgO films, i.e. film thickness, SEM images of cross-sectional view of prepared MgO films for two different W_j at L = 60mm are shown in Fig. 4. According to these results, with increasing W_j, deposition rates (or film thickness) of prepared films become high. When W_j = 4 kW, film thickness is about 300 μm. Namely, deposition rate is 75 μm/min.

So far, we have confirmed that, under the present W_j, MgO films are well prepared when substrates are set in the upstream region, i.e. L_s = 40 - 60 mm. On characteristics of sprayed films, porosity is an important property for some applications.

Figure 5 shows a set of XRD patterns and SEM images of the prepared films at two different substrate positions, (a) and (b) at L_s = 60 mm, and (c) and (d) at L_s = 40 mm. Porosity is estimated from the SEM images of the cross section of the films, i.e. (b) and (d). As is shown clearly, porosity of the film at L = 40 mm is lower compared with the film at L = 60mm. They are about 5 % and 11 %, respectively. Porosity depends strongly on W_s, Ls and combination within Q_f, Q_m and W_j. On the other hand, porosity of MgO films by using water-stabilized plasma is beween 5-10 vol % (measured by using mercury intrusion porosimetry, Archimedean weighing and helium pycnometry)[8]. Roughly speaking, porosity of the present film is nearly the same as that of the film prepared by using water-stabilized plasma with high power ($>$50 kW). Then it is confirmed that MgO films are well prepared by using the gas-stabilized plasma with low power. These coatings can be used for some applications. However, the porosity is too high for the superconducting applications and some additional pore control would have to be used.

According to the previous results [1, 4], with decreasing P_t, plasma jet expands and its velocity and temperature increase greatly in the downstream region. On spray coatings of Al_2O_3 [9] and Mo [10] (melting points are 2015 ℃ and 2620 ℃, respectively), with decreasing P_t, porosity becomes low.

To prepare dense films, therefore, spraying of MgO under low pressure is also done. But MgO coatings are not well prepared under low pressure ($P_t$$<$760 Torr). With decreasing P_t, residence time of powders in plasma jet becomes short as plasma jet velocity in creases. MgO powder may be not heated up to melting point with the present W_j.

In plasma spraying, injected powders should be sufficiently melted and collided to the substrate with high speed. In the future, by decreasing pressure and by increasing jet power, we attempt to prepare dense MgO coating.

4. Conclusions

By using the thermal plasma reactor composed of the forced constricted type plasma jet generator with the feed ring, spraying of MgO films is studied. MgO coatings are prepared for the first time with gas-stabilized plasma jets under certain spraying conditions. Porosity of the films is the same as that of the films prepared by using water-stabilized plasma. It is also confirmed that MgO coating is very sensitive to plasma conditions. In the future, optimization of the processing conditions for more dense MgO coating is studied under various conditions, including high jet power and low pressure.

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Figure Captions

Fig.1. Schematic drawing of the plasma jet reactor system.

Fig.2. 2. X-ray diffraction (XRD) patterns of raw materials, (a), and a prepared film, (b). Symbol ○ is a spectrum of MgO, and Mo is that of molybdenum, i.e. substrate material. Experimental conditions are as follows: Plasma gas (Ar) flow rate Q = 20 *l*/min, feed gas (Ar) flow rate Q_f = 6 *l*/min, MgO powder flow rate Q_m = 0.5 g/min, pressure in the reactor P_t = 760 Torr, jet power W_j = 4 kW, substrate position L_s = 40 mm and spraying time t_s = 4 min.

Fig.3. XRD patterns of the prepared films at four different substrate positions. Symbol ○ is MgO, and Mo is molybdenum substrate. Experimental conditions are as follows: Q(Ar) = 20 *l*/min, Q_f (Ar) = 6 *l*/min, Q_m (MgO) = 0.5 g/min, P_t = 760 Torr, W_j = 4 kW and t_s = 4 min.

Fig.4. SEM images of cross section of prepared films. (a) W_j = 3 kW and (b) W_j = 4 kW, under the conditions that P_t = 760 Torr, Q(Ar) = 20 *l*/min, Q_f (Ar) = 6 *l*/min, Q_m (MgO) = 0.5 g/min, L_s = 60 mm and t_s = 4 min.

Fig.5. XRD patterns of the prepared films and corresponding SEM images for two different substrate positions, i.e. (a) and (b) at L_s = 60 mm and (c) and (d) at L_s = 40 mm. Other experimental conditions are as follows: P_t = 760 Torr, Q(Ar) = 20 *l*/min, Q_f (Ar) = 7 *l*/min, Q_m(MgO) = 0.5 g/min, W_j = 4.5 kW and t_s = 4 min.

Table 1 Experimental conditions and others

Experimental conditions	Value
Chamber pressure P_t	100 – 760 Torr
Jet power W_j	3 – 5 kW
Working gas flow rate Q	20 *l*/min (Argon)
Feed gas flow rate Q_f	4 – 10 *l*/min (Argon)
Powder flow rate Q_m	0.2 – 1.0 g/min
Powder mean diameter	40 μ m
Substrate and its size	Molybdenum plate (15 mm diameter and 0.3 and 1 mm thickness)
Substrate position L_s	30 – 120 mm
Spraying time t_s	4 min
Deposition rate	50 – 80 μ m/min
X-ray target material	Cupper
X-ray intensity	40 kV, 40 mA

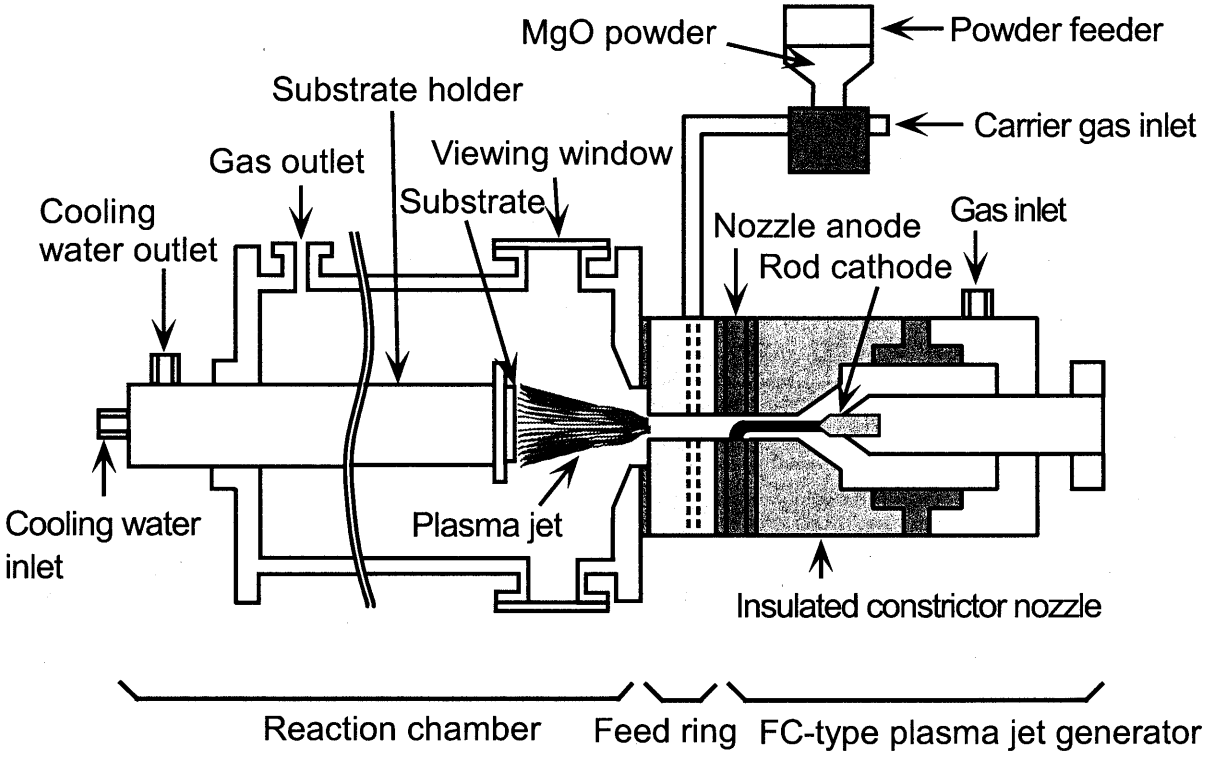


Fig. 1 O. Fukumasa

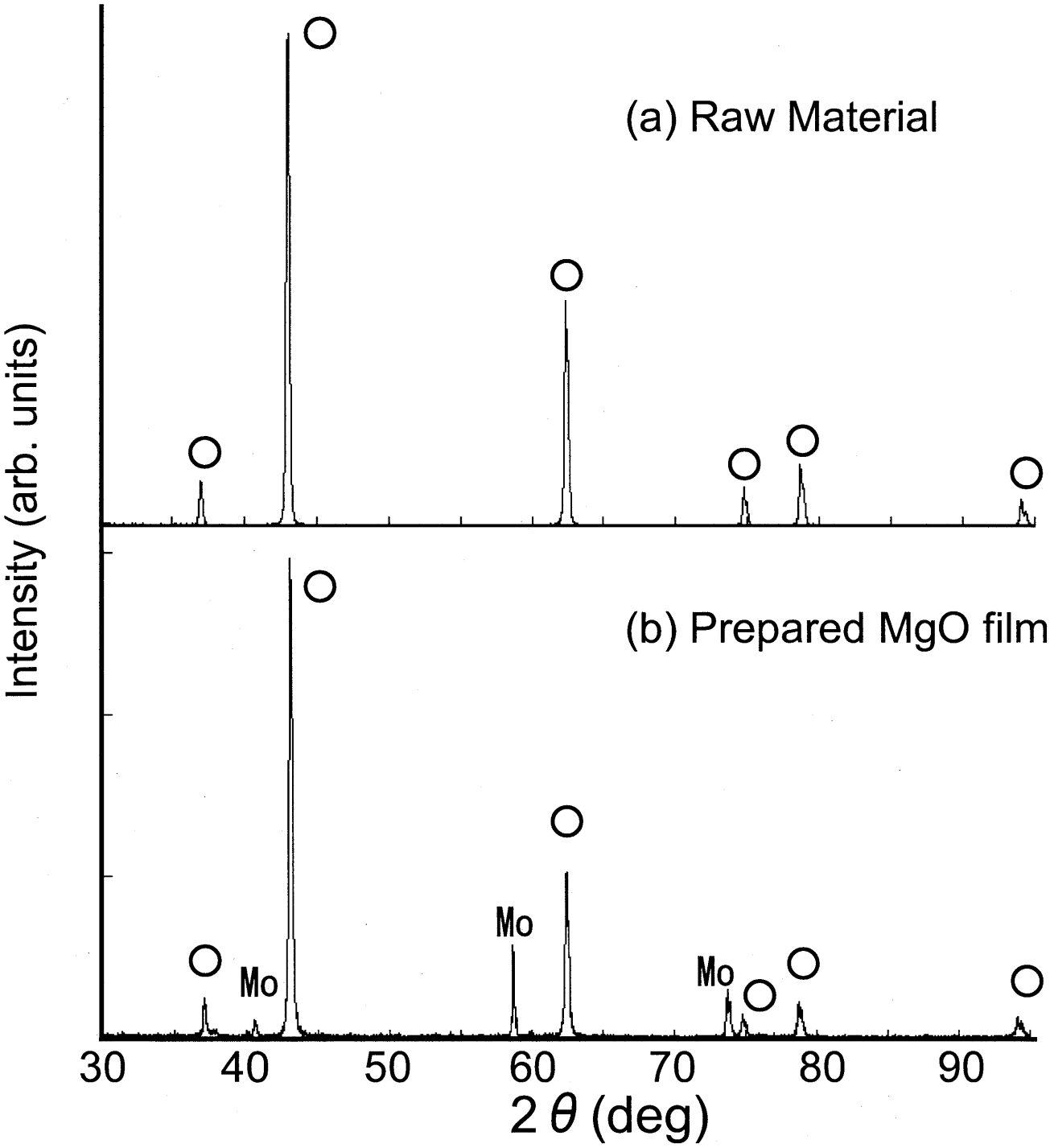


Fig. 2 O. Fukumasa

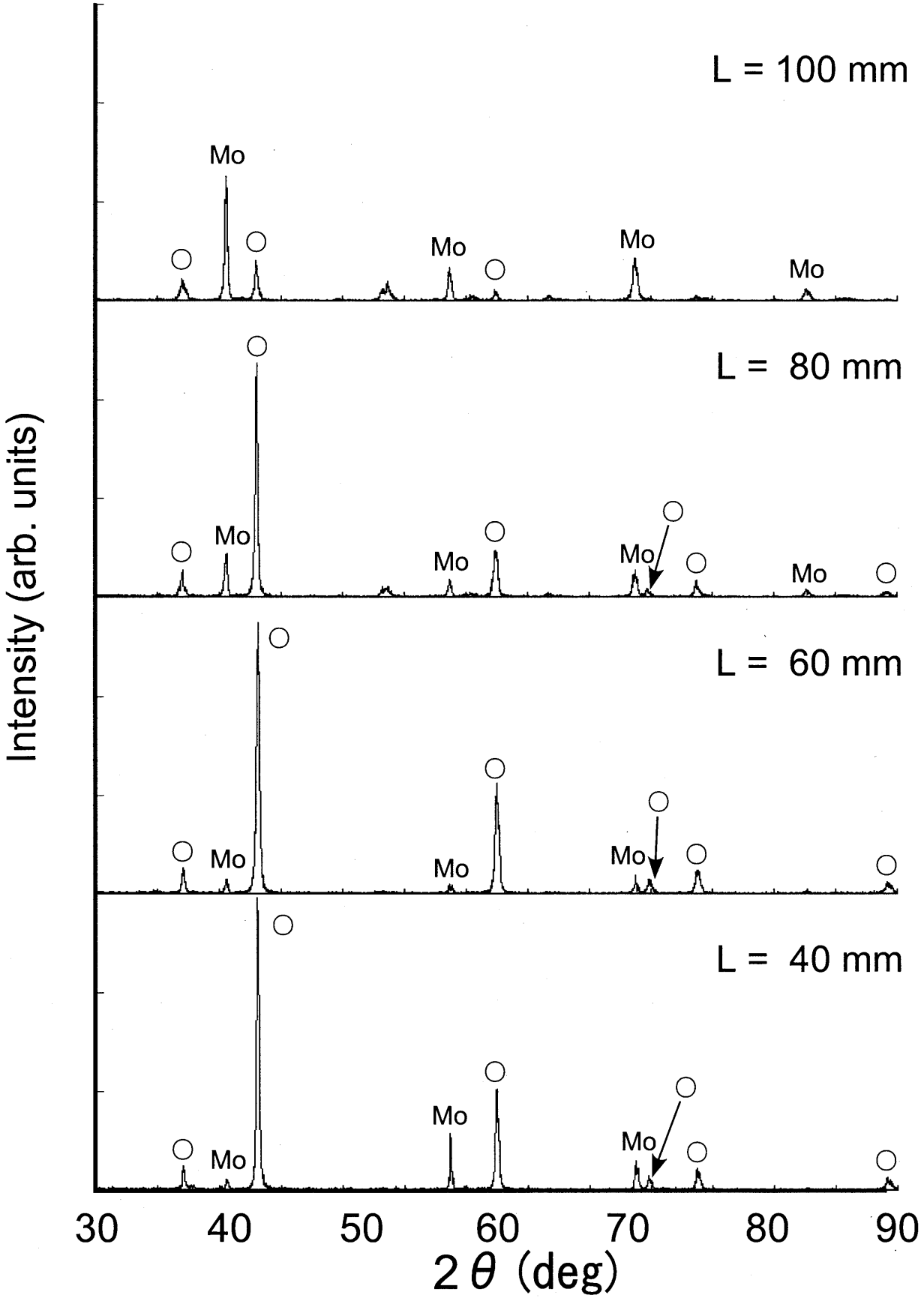
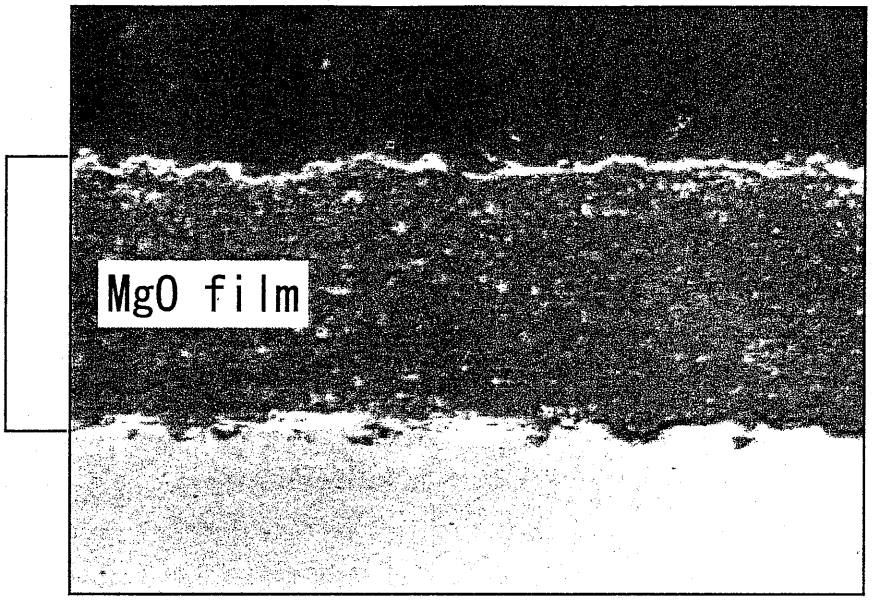
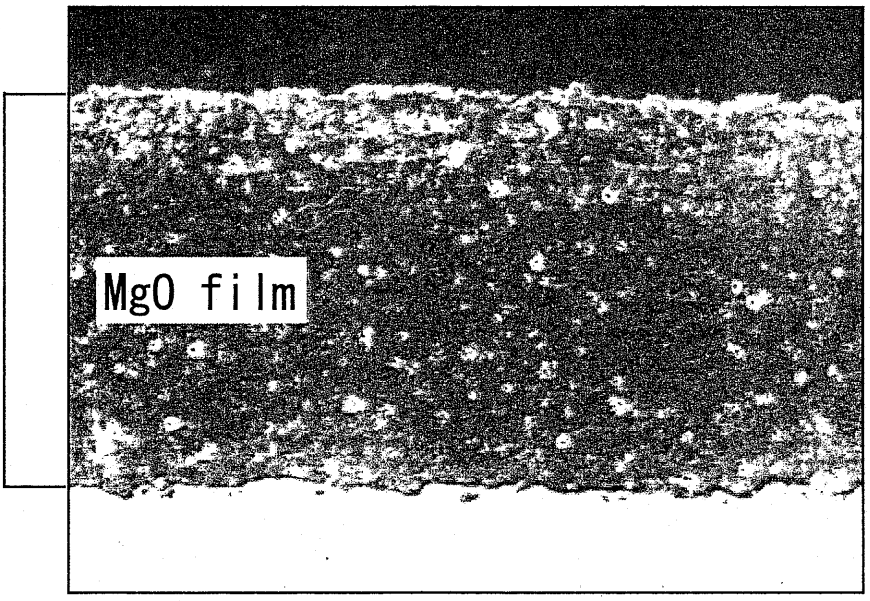


Fig. 3 O. Fukumasa



(a) W_j = 3 kW

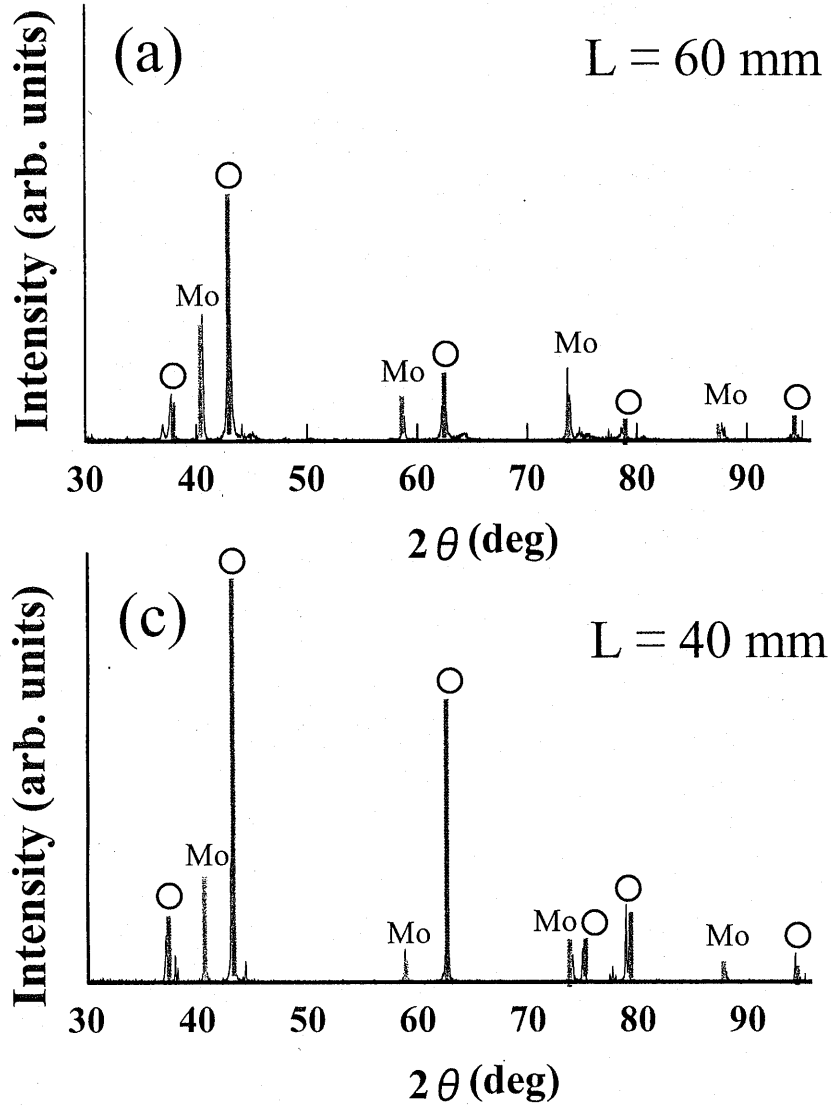


(b) W_j = 4 kW

Fig. 4 O. Fukumasa

XRD patterns

SEM images



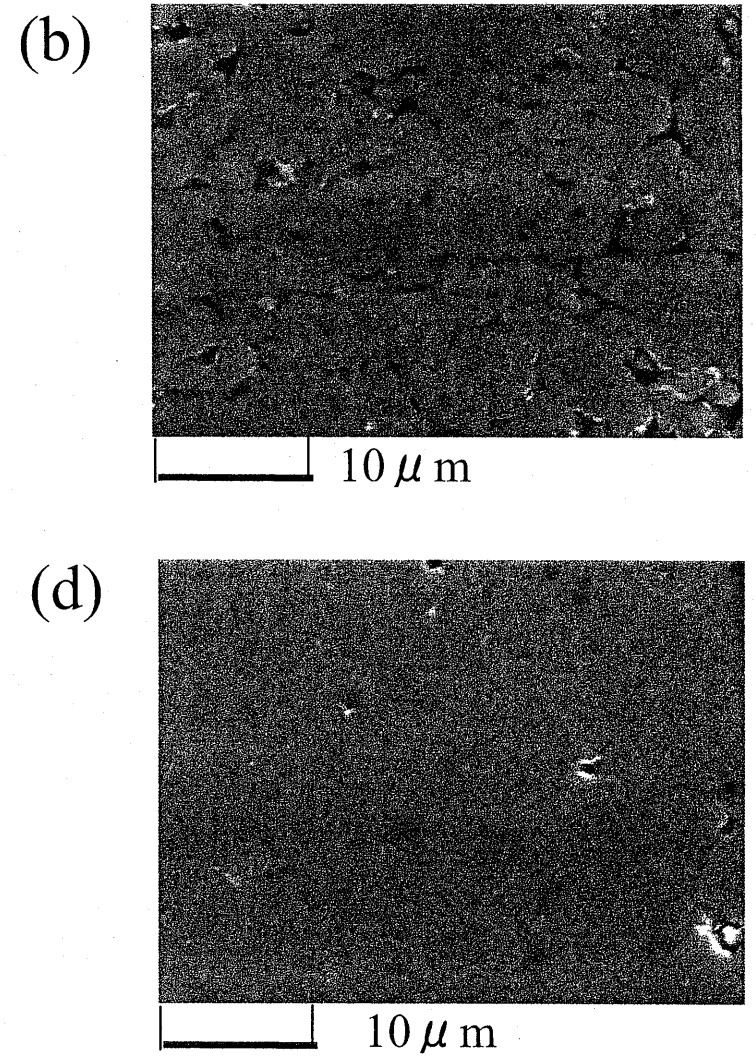


Fig. 5 O. Fukumasa