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AN OVERVIEW OF CVD PROCESSES

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AN OVERVIEW OF CVD PROCESSES

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August 1, 1986

I. Introduction

Recent progress on CVD has been reviewed by Sugiyama¹⁾ and Motojima²⁾. There have been a number of reports on modeling and new processes of CVD at the International Meeting on CVD³⁾. /130*

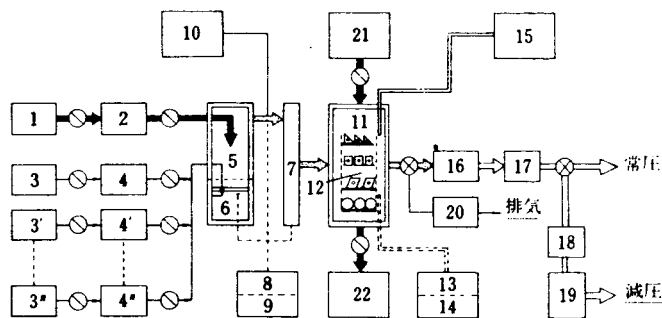
In this review, CVD processes under normal or reduced pressures are examined critically, since they are now in practical usage. Several problems, which arise in much broader applications, are pointed out. New processes of CVD are also described. Superior characteristics of CVD are taken into account and they are applied in practical use.

II. Basic CVD Process

The most basic CVD method is as follows:

First, vapors of metallic or nonmetallic compounds are sent to surfaces of base materials. Then, deposits result from the vapors on surfaces by chemical reactions at a high temperature. All new CVD processes are based on this basic process. In Fig. 1 the flow sheet is shown for the basic process under normal or reduced pressures.

*Numbers in margin indicate foreign pagination.



- | | | | | |
|---------|-----------------|--------------|-----------------|---------------|
| 23 → | めっき試薬, CVD素材の輸送 | 1. CVD試薬 | 9. 同上温度調節器 | 17. 逆流止め用トラップ |
| 24 ⇨ | めっきベーパーの流れ | 2. 分注器 | 10. ベーパー濃度計 | 18. フィルター |
| 25 ⇨ | 使用済みベーパーの流れ | 3. ガス源 | 11. 反応器 | 19. 減圧用ポンプ |
| 26 → | キャリアガスの流れ | 4. 流量計 | 12. 被処理素材 | 20. 排気用ポンプ |
| 27 ···· | VTの加熱と温度調節 | 5. 気化器 | 13. VT用加熱器 | 21. 素材供給器 |
| 28 ···· | ATの加熱と温度調節 | 6. VT中のCVD試薬 | 14. 同上温度調節器 | 22. 製品回収器 |
| 29 ⊙ | バルブ (開閉, 減圧) | 7. ガスマキサー | 15. 反応器内圧力計 | |
| 30 ⊗ | バルブ (切替え, 三方) | 8. VT加熱器 | 16. 反応済みベーパー回収器 | |

Figure 1. Flow sheet of basic CVD process

- | | |
|------------------------------------|--|
| 1. CVD reagent | 16. collector for the reacted vapors |
| 2. fractionator | 17. trap |
| 3. gas source | 18. filter |
| 4. flow meter | 19. vacuum pump for exchanged gases |
| 5. vaporizer | 20. vacuum pump for exhausted gases |
| 6. CVD reagents in the VT | 21. supplier of base materials |
| 7. gas mixer | 22. recovery system |
| 8. VT heater | 23. transport of reagents and base materials |
| 9. thermistor | 24. flow of plating vapors |
| 10. concentration meter for vapors | 25. vapor flow after the reactions |
| 11. reactor | 26. carrier gas flow |
| 12. base materials | 27. VT heating and temperature control |
| 13. VT heater | 28. AT heating and temperature control |
| 14. thermistor | 29. bulb (open and close, reduced pressure) |
| 15. manometer for the reactor | 30. bulb (change of flow directions, 3 ways) |

Materials used in CVD processes include alloys of approximately 38 different elements. These alloys are comprised of various materials such as carbides, nitrides, oxides, borides, silicides and arsenides, etc. In general, they have many excellent properties such as superior hardness, high melting point, nonrust, electroconductivity, and optical transparency. Further, their films can be formed in epitaxial growth, single or multiple crystals, or in amorphous states depending on the CVD conditions. The CVD is not only a method of plating, but also can be applied to whisker, fiber, powder productions, and forming of hard-to-work materials. Its strong penetration power can be utilized to fill voids and for bonding treatment, and syntheses of pure metal or nonmetallic compounds. The application of CVD technology is abundant.

III. Starting Materials and Related Instruments

Halogenated compounds are in general used for plating reagents. Plating is done at 500 - 1400°C and whole materials are heated. When materials require a low temperature treatment (below 400°C), carbonyl, organic compounds, and hydrates are used. They should be highly pure and deposited at lower temperatures than the melting point of the base materials. It is also important that their vapors should be inactive until they reach the surfaces of base materials. /131

Since most of CVD reagents are active, they might react with air. They should be handled with caution, since some of them are

explosive and poisonous. The reagent gases are mixed with carrier gases and transported by them to surfaces of base materials. Monatomic gases such as hydrogen, nitrogen, and argon, and reactive gases such as hydrocarbon and carbonoxide gases are used as carrier gases. Their purity should be as high as 99.99% and they should be dry (saturation point = -70°C).

There are two characteristic temperatures in CVD. One is VT, at which temperature reagents are vaporized. The other one is AT, which is applied to the materials so as to induce CVD reactions.

The VT ranges from minus to 200°C , depending on the phases of reagents at the room temperature. As a temperature bath a regular jar or oil bath can be used. The AT is in general about $400 - 1200^{\circ}\text{C}$ depending on the reagents. In CVD, AT is much higher than VT contrary to the PVD. This yields high quality films and strong bondings. However, high temperatures cause deformations and decomposition of base materials. Therefore, CVD base materials should be thermally resistant.

Such high temperature processes affect designs of CVD and mass production and are one of the major faults of CVD. To overcome this difficulty of the adaptation of base materials to CVD processes, new developments of base materials, and new processes at lower temperatures are investigated. Shikata⁴⁾ has shown the relationship between various CVD methods to produce Si N films and process temperatures in Figure 2.

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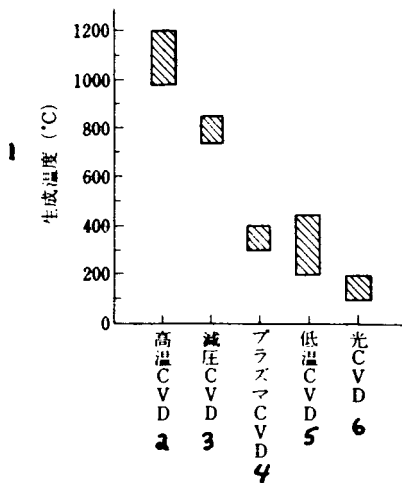


Figure 2. Production temperature of Si₃N₄ by various CVD methods.

1. Production temperature
2. High temperature CVD
3. Reduced pressure CVD
4. Plasma CVD
5. Low temperature CVD
6. Light CVD

The temperatures of VT and AT are directly related to the growth rate of films and crystal conditions. In order to produce a uniform film thickness and good film quality, it is important that the temperature and vapor composition inside the reactor should be maintained uniformly. Other factors which affect the plating rate are types of reagents and plating base materials, flow rates of carrier gases, inner structures of the reactors, position of the base materials in the reactor, vapor pressure, dynamic and static holding positions of base materials.

There are two AT heating methods. One is an external heating method in which the reactor containing all materials is surrounded by a heater. This method's advantage is that any shape of material can be heated uniformly and that the whole material can be treated uniformly. Further, the temperature is accurately controlled by a simple thermistor and mass production is possible. Therefore, this is the most widely used method of all. It is often called a hot-wall type, since the temperature of the reactor wall becomes high. The disadvantage of the method is that efficiencies of thermal energy and reagents are low. The other method is an inner heating type, in which base materials are heated up to high temperatures. Traditional methods such as electrical or high frequency heatings belong to this type. Recent methods such as light and electron beam heatings, plasma heatings, and radiation heatings are also in this type. The traditional methods require special shapes and types of base materials, and the temperature is hard to control. However, the temperature in the reactor is relatively low, and therefore efficiencies of energy and reagents are high. Besides a vaporizer, a reactor, a VT or AT heater, a temperature controller, and a thermistor, the following instruments are necessary to the CVD process:

(1) Fractionator: Since some of the CVD reagents are reactive, they might change their qualities when exposed to the air. They are also harmful if the workers are in contact with

the reagents. Further, equipment may rust by the reagents. Therefore, it is recommended to measure the necessary amount of reagents without exposing them to the air. Interested readers can consult the present author's patent ⁵⁾ .

(2) The flow meter controls flow rates of the carrier gas for monatomic gases. It is connected to the gas bulb of the commercial high pressure gas container. The flow rates of plating vapors or compositions of gas mixtures in the reactor are heavily dependent on the flow rate of carrier gases. Thus, highly accurate flow meters should be used.

(3) The concentration meter measures partial pressures of various components in a mixture of plating vapors and carrier gases. It also adjusts a composition of the mixture. Thus, use of a highly accurate and reproducible meter is recommended.

(4) The supporter holds, vibrates, or rotates plating materials. A supplier and recovery system are also needed; a mass production system is especially considered, whether continuous or semicontinuous.

(5) Other equipment includes a vacuum pump which evacuates air from the CVD system, a manometer which measures pressures in the reactor, especially when air is substituted by hydrogen

Table 1. Commercial CVD equipment and its characteristics.

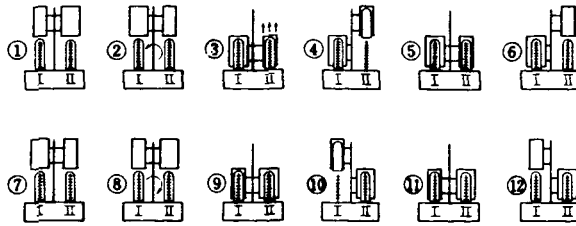
1	メーカー名	2	国名	3	加熱法	4	圧力	5	反応器の最大内容積
6	ベルネックス	7	スイス	8	外熱式	9	減圧		380mm ϕ × 1100mm
10	ツール・メタル		"		"		"		—————
11	S I C	12	アメリカ		"	13	常圧		450 × 700
14	プランジェ	15	オーストリー		"		"		360 × 900
16	シグマコーティング	17	日本		"		減圧		—————

- | | |
|--|-------------------|
| 1. manufacturer | 14. plunge |
| 2. country | 15. Austria |
| 3. heating system | 16. sigma coating |
| 4. pressure system | 17. Japan |
| 5. maximum inner volume in the reactor | |
| 6. Belnex | |
| 7. Switzerland | |
| 8. external | |
| 9. reduced pressure | |
| 10. tool metal | |
| 11. SIC | |
| 12. United States | |
| 13. normal pressure | |

gases, and, at the time of reaction, a cooling trap for unreacted reagents and reaction products, a trap which collects air outside the reactor when it is flowing into the system, and a vacuum pump and filters which are used only for the low-pressure CVD.

IV. CVD system

These days the above-mentioned instruments and parts,



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| ①-Ⅱ, ②-Ⅱ : CVD完了, 炉を引上げる | ⑦-Ⅰ, ⑧-Ⅰ : CVD完了, 炉の引上げ。 |
| ①-Ⅰ, ②-Ⅰ : 反応器内に素材をセットし,
炉と冷却器の位置を交換する。 | ⑦-Ⅱ, ⑧-Ⅱ : 炉と冷却器の位置を交換。 |
| ③-Ⅰ, ④-Ⅰ : CVD開始 | ⑨-Ⅰ, ⑩-Ⅰ : 冷却器を下げ反応器を冷却。
冷却器と反応器を上げ, 器内
の製品を回収する。 |
| ③-Ⅱ, ④-Ⅱ : 反応器Ⅱの急冷を開始し, 反
応器と冷却器を引上げて製品
を回収する。 | ⑨-Ⅱ, ⑩-Ⅱ : 炉を下げて, CVD開始。
CVD続行。 |
| ⑤-Ⅰ, ⑥-Ⅰ : CVD続行 | ⑪-Ⅰ, ⑪-Ⅰ : 冷却完了, 冷却器を上を上げ
る。 |
| ⑤-Ⅱ, ⑥-Ⅱ : 冷却器と反応器を下げ, 冷却
器をもとして反応器をセット
する。 | ⑫-Ⅱ, ⑫-Ⅱ : CVD完了, 以下1-Ⅱに続く。 |

Figure 3. CVD recycling process which consists of two reactors.

1. CVD has been done. The heater is lifted.
2. Base materials are placed in the reactors and the position of the heater and cooler are exchanged.
3. CVD is started.
4. The reactor II is quenched. The reactor and the cooler are lifted and the products are recovered.
5. CVD is continued.
6. The cooler and reactor are set in place.
7. CVD has been done.
8. Interchange of the heater and reactor positions
9. The cooler is lowered and the reactor is cooled down. The cooler and reactor are lifted and the products in the reactors are taken out.
10. The heater is lowered and the CVD is started.
11. The cooling has been finished and the cooler is lifted.
12. CVD has been done, and continues to 1-II.

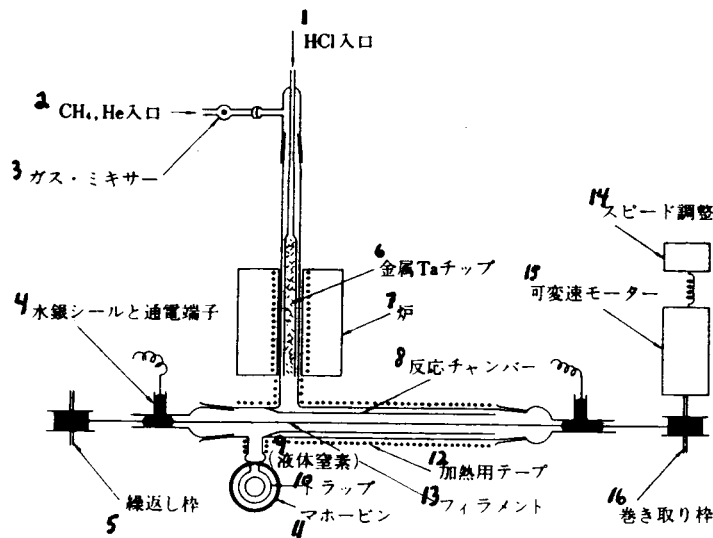


Figure 4. CVD process for thin wires.

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|------------------------------------|----------------------|
| 1. HCl inlet | 9. liquid nitrogen |
| 2. CH ₄ and He inlet | 10. trap |
| 3. gas mixer | 11. jar |
| 4. mercury seal and electric inlet | 12. tape heater |
| 5. repeating frame | 13. filament |
| 6. metal Ta chips | 14. speed controller |
| 7. heater | 15. variable motor |
| 8. reaction chamber | 16. collector frame |

starting reagents such as CVD reagents and gases are easily available in Japan. The CVD system can be constructed following Fig. 1. It is noted that the system should be air-tight, since the reagents and gases are often harmful and explosive. In recent years, one can get commercial CVD systems which are a set of the CVD system and related instruments. In Table 1, some of

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them are listed. All of them are the widely used external heating type. In Fig. 3, the CVD recycling process system developed by Belnex, Inc.⁶⁾ is shown.

This system consists of two reactors, a heating system, and a high speed air cooling system. It uses thermal energy efficiently, working hours become short, and it is adequate for mass production.

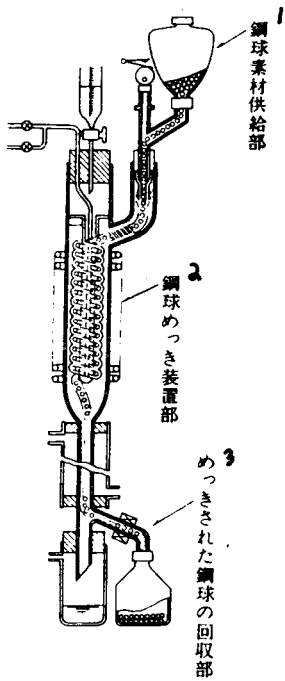
V. Mass Production Process of CVD

One of the main reasons to use CVD is to increase productivity. Since the old CVD used an array or batch system, its productivity was low. In order to improve productivity, the CVD process must have a continuous or semi-continuous system. For wires, films, and strips whose shapes are regular, the continuous process has been done. In this case electrical heating is used. Mercury is used as an electrode and as a seal. The system is displayed in Fig. 4.

The plating system is enclosed in this case. It is quite new and reported by W. J. Hefferman et al.⁷⁾ When base materials have uniform diameters such as sphere and bearings, they are processed continuously by a system shown in Fig. 5⁸⁾. The system uses either an external or high-frequency internal heating system.

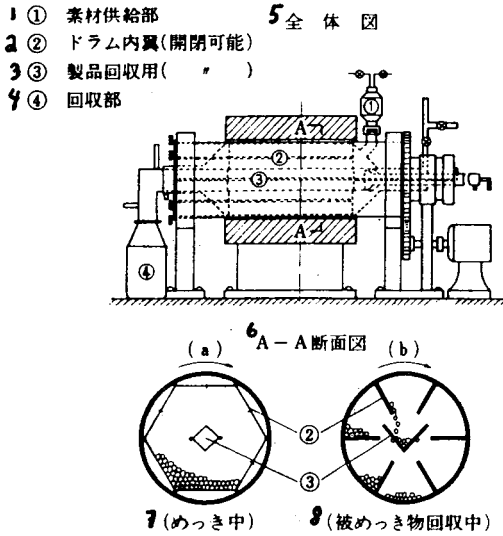
The process is shown in Fig. 6⁹⁾. It has a barrel in the

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1. steel ball supplier
2. steel ball plating
3. recovery system

Figure 5. Continuous CVD process for steel balls.



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Figure 6. Semi-continuous CVD system for various shape materials.

- | | |
|--------------------------------------|-----------------------|
| 1. base material supplier | 5. whole system |
| 2. drum wing (open and close) | 6. A-A dissection |
| 3. product recovery (open and close) | 7. plating processing |
| 4. recovery | 8. product recovery |

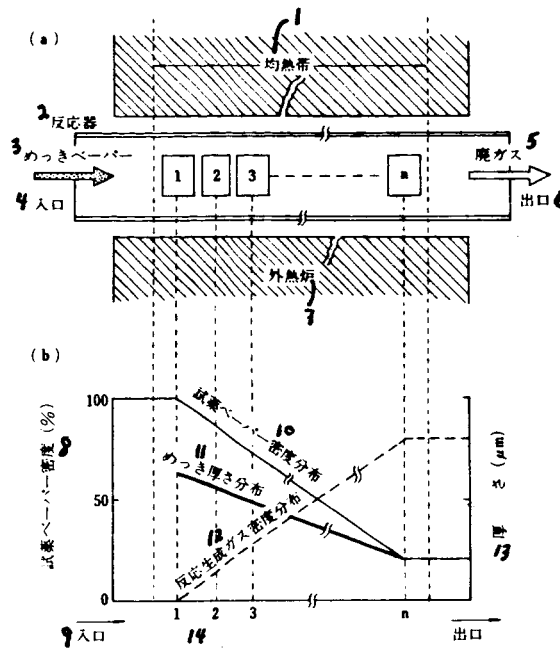


Figure 7. Position (a) of base materials arranged in an array in the reactor and densities and thickness distribution of reagent and resulting gases along the flow of plating vapors.

- | | |
|------------------------------|---|
| 1. constant temperature zone | 8. vapor density |
| 2. reactor | 9. inlet |
| 3. plating vapor | 10. reagent vapor distribution |
| 4. inlet | 11. thickness of film |
| 5. exhausted gases | 12. resulting gas density distribution |
| 6. outlet | 13. thickness |
| 7. external heater | 14. position of CVD base materials in the reactor |

CVD reaction chamber. It processes semi-continuously with the external heating system, if base materials are uniform. It is especially adequate for mass production of various small sizes of chips, bolts, nuts, taps, drills, etc. The base materials are

sent to the rotating barrel and recovered from it. These processes are semi-continuous, but the CVD process itself is continuous.

VI. Uniform Thickness Process for Films

When base materials are placed in an array as shown in Fig. 7(a) for the flowing CVD process, the concentrations of reagent vapors decrease as the vapors flow through the reactor. Fig. 7(b) shows the situation. In this case the thickness of a yielded film decreases from the inlet to the outlet. There is a possibility that film quality may vary, too. High quality products cannot be produced in this way. Similar results are expected for a batch process. /135

The newly-developed CVD process with an inner heating system improves such defects. The process is shown in Fig. 8. In this process system, the reactor (A) is divided by the two walls (E and F). The heater (H) is placed at the center. When the heater is on, the two temperature regions appear. One is for a vaporization zone (VT) (B); the other for a reaction zone (AT) (C). Base materials (D) are located in the AT region near the heater. They are heated in the AT. Plating vapors pass through the VT zone and enter into the AT zone through the first wall which has many holes. They reach the surfaces of base materials and CVD reactions take place. 10)

Since the resulting gases prevent the reactions, they are removed from the holes of the second wall. The inner heating

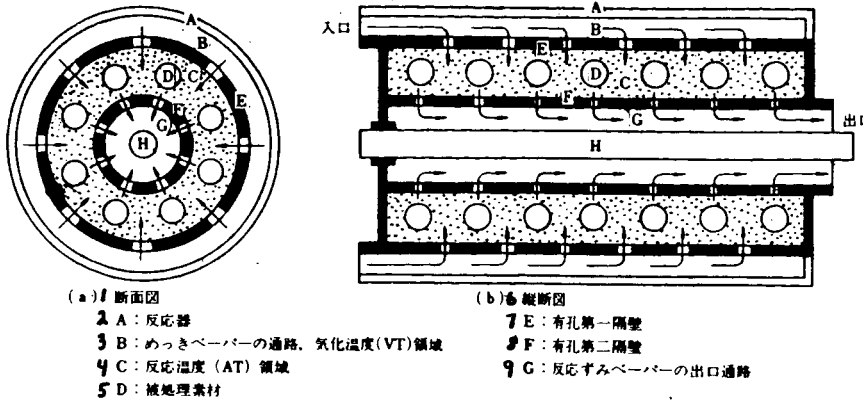


Figure 8. Principle of CVD process: inner heating system.

- | | |
|---|---------------------------------|
| 1. lateral section | 7. the first wall with holes |
| 2. reactor | 8. the second wall with holes |
| 3. passage for plating vapors
vaporization temperature (VT) zone | 9. passages for unreacted gases |
| 4. vaporization temperature (AT) zone | |
| 5. base materials | |
| 6. longitudinal section | |

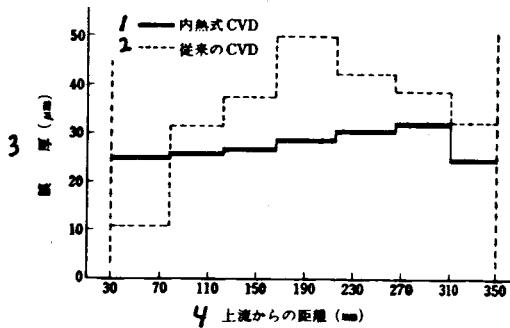


Figure 9. Comparison of film thickness.

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|--------------------------|
| 1. inner heating CVD |
| 2. external heating CVD |
| 3. thickness |
| 4. length from the inlet |

CVD process yields more uniform films than the traditional external CVD method. A comparison of the two methods is shown in Fig. 9.

The author developed a combination system of the inner heating CVD process and a barrel type. It is displayed in Fig. 10. It yields uniform thickness and good quality films. Also mass production is possible.

VII. CVD Method and Precision Control of Film Thickness

The traditional CVD method can produce films with limited accuracy of thickness. Plunger and bearing supporters of a fuel pump require 10^{-2} to 10^{-3} precision. These high accuracy film controls can be attained by a system shown in Fig. 11(a)¹¹⁻¹³).

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The process is as follows: Two parts are separated by a clearance which is a sum of the film thickness of the two. CVD is performed at this stage by using super hard material CVD. Then the surfaces of the two are covered with super hard materials simultaneously¹⁴). If the CVD process proceeds, the clearance is filled with film deposit. However, if one part is fixed while the other is vibrated along its axis or rotated around the axis, the momentum drops when the CVD proceeds and film thickness becomes close to the desired one. Thus, the film thickness is controlled by detecting this momentum change. In our vibration experiment, the initial acceleration rate and amplitude of vibration of 50 Hz were 48 g and 60 μm , respectively. When they dropped to 36 g and

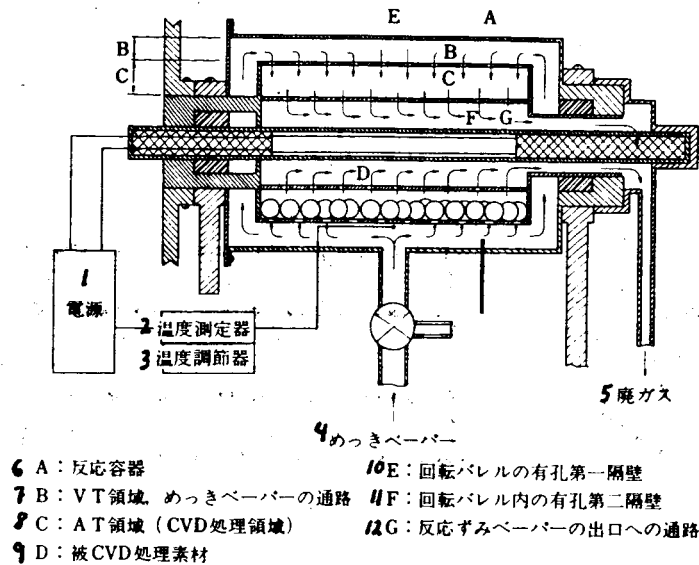


Figure 10. CVD mass production system with inner heating and barrel methods.

1. electric source
2. thermometer
3. thermistor
4. plating vapor
5. exhausted gases
6. reactor
7. VT zone, plating vapor passage
8. AT zone (CVD treatment)
9. base materials
10. the first wall with holes in the rotating barrel
11. the second wall with holes in the rotating barrel
12. passage of exhausted gases

30 μm , the resulting TiN layer was 24 μm thick. The crystal particles in the film are very fine and film surfaces are near mirror quality and flat. The surface roughness is about 2S and it is unnecessary for the post-polishing treatment. Various nozzles and

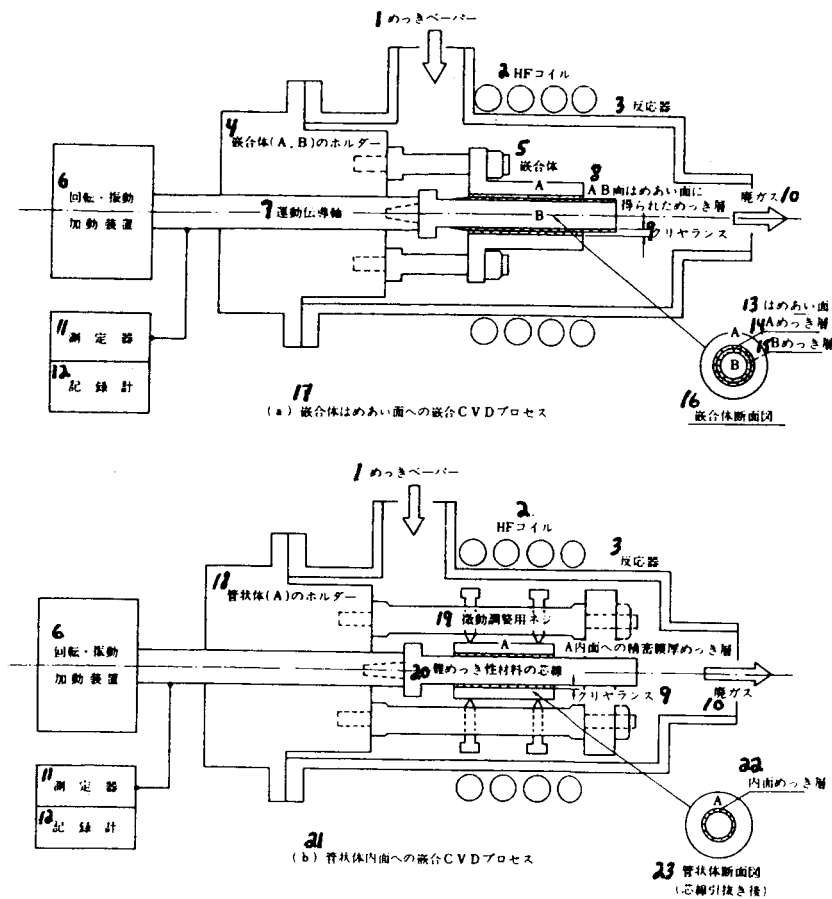


Figure 11. CVD process for joints.

- | | |
|------------------------------------|--|
| 1. plating vapor | 15. B plating layer |
| 2. HF coil | 16. lateral section |
| 3. reactor | 17. CVD process for joint plane |
| 4. joints (A, B) holder | 18. cylinder holder |
| 5. joint | 19. nuts for fine tuning |
| 6. rotation or vibration generator | 20. axis insides of the cylinder |
| 7. axis | 21. (b) joint CVD process for cylinder |
| 8. plating layers on A and B | 22. inner plating layer |
| 9. clearance | 23. cylinder lateral section |
| 10. exhausted gases | |
| 11. meters | |
| 12. recorder | |
| 13. joint lateral section | |
| 14. A plating layer | |

cylinders whose diameters require a high precision may be obtained by a modification of the above method; movable mandrel materials should be of CVD type, and their diameters made with high precision. These become an axis cylinder. Fig. 11(b) is a schematic diagram of this method¹⁵⁾. This process can yield accurately controlled film thickness on both sides or on one side of hardly processed materials for which the post-process is not required. However, it is difficult to do mass production. Because of its special values, basic experimental tests are performed at Kikaigiken^{16, 17)}. /137

VIII. Low Temperature CVD and New Process

As mentioned previously, the traditional CVD has the defect that whole base materials have to be heated up. To lower the temperature various new processes have been proposed as shown in Fig. 2. Among them are MOCVD which utilizes organic metals or hydrides which have low-depository temperatures, plasma CVD, light CVD which used thermal reaction, and photo decomposition.

(1) MO-CVD (Metalorganic Chemical Vapor Deposition)

This follows the basic principle of the CVD process. The CVD reagents used in the MOCVD are those whose deposition temperatures are low, such as alkyl compounds or hydrides. Thin layers are formed on base materials at a low deposition temperature. This process has originally been reported for crystal growth technology for GaAs. The GaAs crystal has twice as fast

an access time as that of Si. Later various crystal mixtures of III - V and II - VI group compounds were found to be produced by this method. Now they are going to be applied for computer chips which require a high speed access time. However, the method is still in an infant stage and there remain many problems to be solved.

(2) Plasma CVD (Plasma Enhanced CVD)

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In the plasma CVD, a plasma region is created in the reduced reactor, base materials are placed at the region into the reactor and heated at a relatively low temperature, and CVD plating vapors are introduced in the reactor. The vapors generate various gases by chemical reactions due to non-equilibrium plasma, and CVD films are formed on the surface of base materials. In this case high energies due to plasma enable thermodynamically unfavorable reactions to proceed. Also, some slow reactions are enhanced. The plasma CVD can be performed at low temperatures and yields functional, amorphous, and super hard films. In electronics, Si_3N_4 films are applied to passivation films of MOS semidiodes. Further, they are expected to be applied to LSI and super LSI. Amorphous Si films may be utilized as solar batteries. SiO_2 and GaAs are used for making core parts of clad crystal tubes.

(3) The light beam CVD process can form a film locally by just heating that part of the material by laser, electron, or

or solar beams.

Of them, L-CVD (Laser CVD) has been investigated most. The L-CVD has two processes: one is a local heating, the other, due to laser photo-excitation chemically, promotes the deposition rate. The process has two branches. The first one is to decompose or dissociate excited atoms to molecules by laser beams. The second is to promote chemical reactions at excited states. Since the laser beam is monochrome and highly powerful, it is possible selectively to enhance reactions of resonating atoms or molecules in non-resonance mixtures.

The L-CVD requires low temperatures because of its specificity. The deposition rates can be large and base materials are little damaged. Local plating is also possible. Furthermore, choosing a special wavelength of laser beam, it is possible to form a film of particular atoms or molecules in the mixture. In CVD application, it still requires much basic research.

IX. Summary

It is important to investigate CVD and PVD processes. Bunshan¹⁸⁾ developed the ARE method, which is a combination of CVD and ion-plating. Stimulated by this invention, plasma CVD was discovered, which is a hybrid of CVD and sputtering. More recently, the L-CVD process, which is a hybrid of CVD and laser technology, was investigated. In the future, new processes may be developed by hybrids of various technologies.

I have been retiring my CVD research for 5 years. During this period a number of reports have been published, and all of them are excellent. May I wish further progress in this field.

Finally, but not least, I would like to thank Mr. Shikada at Kikaigiken and Mr. Abe at Kawasaki Taisu, Inc., for sending me a lot of information of the field. Further, I am grateful to the late Mr. Shimamura, manager of material science, who supported the CVD research at the Kikaigiken.

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16. Abstract In this review, CVD processes under normal or reduced pressures are examined critically, since they are now in practical usage. Several problems, which arise in much broader applications, are pointed out. New processes of CVD are also described. Superior characteristics of CVD are taken into account and they are applied in practical use.			
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