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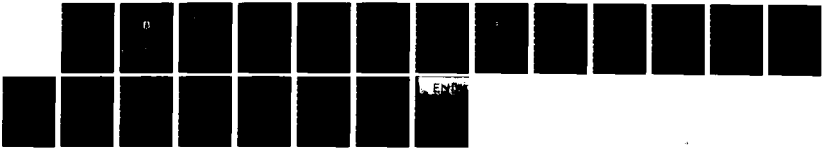

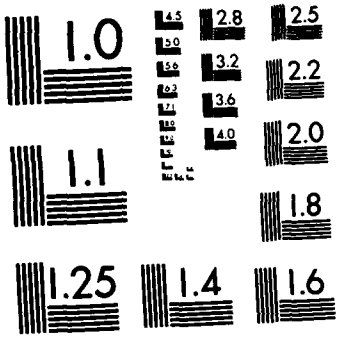

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THE PREPARATION OF LOW-LOSS OPTICAL FIBER WITH MICROWAVE PLASMA ACTIVATED CHEMICAL VAPOR DEPOSITION PROCESS

by

D. Deng, H. Luo, et al

Approved for public release;
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EDITED TRANSLATION

FTD-ID(RS)T-1364-83 29 September 1983

MICROFICHE NR: FTD-83-C-001217

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By: D. Deng, H. Luo, et al

English pages: 15

Source: Tongxin Xuebao, Vol. 3, Nr. 1, 1982, pp. 33-38

Country of origin: China
Translated by: SCITRAN
F33657-81-D-0263
Requester: FTD/TQTD

PREPARED BY:

TRANSLATION DIVISION
FOREIGN TECHNOLOGY DIVISION
WP-AFB, OHIO.

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Editor-in-chief: Editing Committee of China Institute of Communications, 13 West Changan Street

Publisher: People's Post and Telecommunications Publishing Co. 27 West Changan Street

Domestic Distributor: local city level post offices

International Distributor: China International Bookstore (P.O. Box 2820, Beijing)

Printer: Beijing First Printing Plant

Beijing Periodical Registration Certificate No. 579
Domestic Code: 2-676 International Code: Q395
List Price: 0.80 Yuan Published in January 1982

The Preparation of Low-loss Optical Fiber with Microwave Plasma Activated Chemical Vapor Deposition Process /33

Deng Ducai, Luo Huiying, Wang Yanfang, Xiong Jianchun and Wang Shubin
(Wuhan Research Institute of Post and Telecommunications Science)

(ABSTRACT) This paper describes the basic principle and major characteristics of the fabrication apparatus of the microwave plasma activated chemical vapor deposition method. In the meantime, it also describes the basic data of the optical fiber drawn from the preform prepared by this method. The loss has already reached 5dB/km (at 0.85μm wavelength) in the multimode step index optical fiber presently prepared. The minimum loss is 2.48dB/km (at 1.07μm wavelength). The deposition efficiency of the starting material has already exceed 90%.

I. Introduction

The present practical optical communications fibers are quartz fibers prepared by the chemical vapor deposition method (CVD in short). The fabrication techniques can generally be divided into the outside vapor phase oxidation technique OVPO and the inside vapor phase oxidation technique IVPO. The former includes methods such as Outside Vapor Deposition (OVD in short), Vapor Axial Deposition (VAD in short), etc. The latter includes the Modified Chemical Vapor Deposition method (MCVD in short), and the microwave Plasma activated Chemical Vapor Deposition method (PCVD in short) which is discussed in this paper.

The technology of the outside vapor phase oxidation method is very complex. Under ordinary conditions, it is not easy to produce high quality optical fibers. In the inside vapor phase oxidation technology, because the reaction is carried in a tube, it is relatively easier to avoid contamination from the outside environment to produce low-loss optical fibers. In the MCVD

method, a hydrogen and oxygen flame was used as the heat source. It is simple and easy. Therefore, this method is widely adopted in our country.

In the MCVD method, due to the fact that the deposition material temperature reaches 1400°C ~ 1600°C, the gas phase reaction forms oxide powder first and then it was melted. The core layer usually only allows the deposition of approximately 60 layers with various concentrations of impurities to obtain a close to ideal distribution of index of refraction. Therefore, the control of this method is relatively difficult. The consistency is comparatively poor. The bandwidth characteristics are not good. The deposition efficiency is low (usually 15%~30%). /34

As compared to the aforementioned method, the PCVD method[1] has the following advantages: it is not required to use a hydrogen-oxygen flame for heating. The deposition temperature is only close to 1000°C. The control is good. The deposition layer thickness can be less than 1μm. The core layer of an optical fiber can obtain a close to ideal distribution of index of refraction by depositing over a thousand layers with various impurity concentrations. The optical fiber has good geometrical and optical characteristics. Especially, the bandwidth is wide and the reproducibility is good. The deposition efficiency is high, nearly 100%.

Presently, there have been many reports on the successful fabrication of multimode gradient index optical fibers of high quality[2]. It is projected that it will show its unique advantages in the fabrication of single mode optical fibers.

This paper primarily describes the basic principle and the major characteristics of the apparatus used to fabricate optical fibers in the PCVD method. In the meantime, the basic data of the optical fiber drawn from the preform prepared with the apparatus is described. The loss of the multimode step index optical fiber prepared at the present moment is 5dB/km (at the wavelength of 0.85μm). The minimum loss is 2.34dB/km (at the wavelength of 1.03μm). The deposition efficiency has already reached as high as 90%.

II. Basic Principle

The major characteristic of the PCVD method is the use of a non-isothermal plasma (also called a cold plasma) as the reaction source as shown in Figure 1.

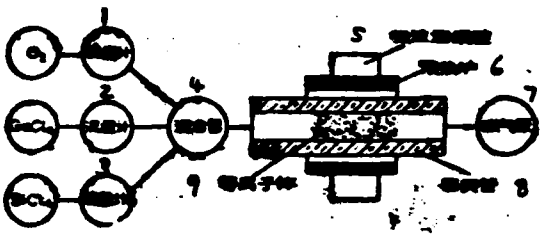

Figure 1. Principle of the PCVD method.

Key:
1. flowmeter
2. flowmeter
3. flowmeter
4. flowmixer
5. microwave resonance cavity
6. pre-heating furnace
7. suction pump
8. quartz tube
9. plasma

After the high purity halides such as $SiCl_4$ and $GeCl_4$ were carried into the reaction tube by the carrier gas O_2, they entered the strong electrical field in the resonance cavity. The gas material was excited and ionized to maintain a glow discharge. A non-isothermal plasma was thus created. The neutral molecules of various materials were ionized into charged particles (electrons, cations, anions) and neutral particles (gas atoms, molecules, excitons, metastable atoms). In the mixture, silicon and oxygen form the compound SiO_2. It was deposited on the inner surface of the reactor tube through diffusion. This is a microwave discharged chemical reaction.

The major characteristic of the PCVD method is that the reaction proceeds at a lower temperature than that of the corresponding thermal reaction. This brings about a series of advantages in the fabrication of optical fibers.

Because the plasma is not limited by the heat capacity of the reactor tube, therefore, the resonance cavity can be moved back and forth at a very fast rate along the reactor tube. Consequently, thousands of layers of homogeneous and transparent thin films can be prepared. Due to the fact that there is no powder process involved, the deposition efficiency is high.

The temperature of the pre-heating furnace surrounding the reactor tube is at around 1100°C. Its function is to maintain the temperature match between the inner wall of the reactor tube and the deposition layer to avoid cracking. In the meantime, it will reduce the chlorine content in the gas bubbles formed in the deposition layer.

The fabrication of optical fibers using the PCVD method can be approximately divided into three stages: the first stage is to deposit a core layer in the reactor tube (which finally becomes the envelope of the optical fiber). The second stage is to heat it up to 1900°C ~ 2000°C using a hydrogen-oxygen flame to melt it down to a preform. The third stage is to reheat the preform in a graphite furnace again to around 2000°C and draw optical fibers under controlled conditions.

III. Experimental Apparatus

The apparatus used to develop the PCVD method is primarily composed of the following components, as shown in Figure 2.

1. Microwave System

The microwave system is comprised of a continuous magnetron tube with 150W maximum output power and working frequency at 2450MHz, a plasma resonance cavity, and the corresponding waveguide elements. /35

According to the requirements of the technology, the adoption of a re-entry resonance cavity was more reasonable. The re-entry type cavity developed used a ring coupled input. Under actual experimental conditions (working pressure 5 torr, input power 150W), glow discharge could be obtained without the excitation of a Tesla coil. The maximum plasma volume produced could exceed 20cm^3. The maximum plasma column length could exceed 20cm. The requirements of the PCVD method were completely met[3].

A 30dB fixed direction coupler was connected in between the microwave cavity and the magnetron tube to monitor power. All the connections were made with 50Ω coaxial cables and N type connectors, as shown in Figure 3.

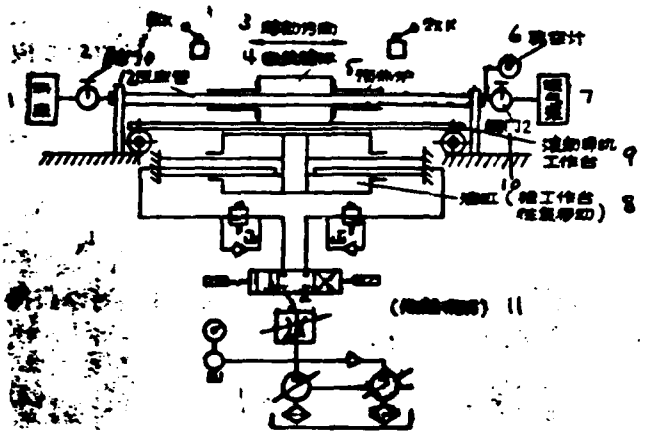

Figure 2. Schematic Diagram of the PCVD Apparatus.

Key:
1. Material source
2. Valve 1
3. Direction of motion
4. Microwave cavity
5. Pre-heating furnace
6. Vacuum gauge
7. Suction pump
8. Oil tank (moving the work platform back and forth)
9. Rolling platform
10. Valve 2
11. Hydraulic system
12. Reactor tube

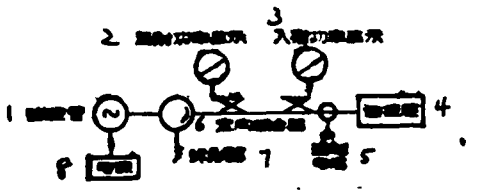

Figure 3. Schematic Diagram if the Microwave System.

Key: 1. Magnetron tube
2. Monitoring reflected power
3. Monitoring incident power
4. Resonance cavity
5. Coaxial cable
6. Fixed directional coupler
7. Spherical device
8. Power supply

2. Gas Handling System

In front of the reactor tube, there are flowmeters corresponding to the various starting materials ($SiCl_4$, $GeCl_4$, O_2, etc.), expansion bottles, and a mixer. In order to prevent the impurities in the air from entering the system, it is maintained at a pressure slightly more positive than atmospheric pressure before valve 1. Behind the reactor tube, there are a suction pump, vacuum gauges, etc. By adjusting valve 2, it is possible to maintain a working pressure of from 1~30 torr in the reactor tube.

3. Reactor

The reactor is composed of the reactor tube, the resonance cavity, and the pre-heating furnace. The reactor tube remained stationary. Its outer diameter is 10mm, inner diameter is 8mm. It is a quartz tube. The water cooled resonance cavity and pre-heating furnace were installed together, and the entire unit was able to move back and forth.

4. The Melting Lathe

In order to realize the linear back and forth motion with infinite speed adjustments between L=0 ~ 80cm, a hydraulic lathe was adopted. Its advantages include: ease to realize the linear back and forth motion, direct drive working mechanism, ability to adjust speed continuously in a relatively large range, stability

of the transfer motion, uniformity of speed, etc. It is easier to obtain a uniform deposition thickness and to realize automatic drive and control. The mechanical structure and electrical circuit are simple. The lifetime is long.

The melting lathe working distance L=0 ~ 80cm, which is continuously adjustable. The traveling speed V=0.4 ~ 20m/min. It is adjustable without discontinuity. The variation of speed is less than 20%. The temperature rise is less than 10°C.

IV. Deposition Experimental Results and Discussion

1. Static Experiments

The resonance and the reactor tube were relatively stationary. The primary objective was to study pure SiO_2 deposition.

The starting materials were $SiCl_4$ and O_2. When the reaction was carried out at room temperature, there was SiO_2 deposition. However, there were large areas of cracks. Furthermore, it peeled off easily after reaching a certain thickness. This was caused by /36 the fact that the reactor tube wall temperature was too low to match with the temperature of the deposit and thus creating stress, as well as by the excessively high chlorine content in the deposition layer. Then the temperature of the pre-heating furnace was increased to over 800°C, the deposit was uniformly thick transparent SiO_2 layer.

The plasma column existed basically symmetric to the center of the cavity. The larger the input power and the lower the working pressure was, the longer the plasma column became. The major deposition part was not located in the center of the cavity. Instead, it occurred away from the center of the cavity toward the gas inlet by a few centimeters. This is because when the materials enter the plasma column immediately react and deposit on the reactor wall. The length of the deposit and the deposition efficiency are primarily determined by the flow rate V_m of the reactant gas in the reactor tube. The flow speed V_m can be obtained from the following equation:

$$V_m = Q/\pi \cdot r^2 \cdot p' \qquad (1)$$

when:
Q is the total flow
p' is the ratio of the working pressure versus the atmosphere pressure
r is the inner radius of the reactor tube.

When the speed V_m is several hundred meters per minute and the reactant gas stays in the plasma column for several milliseconds, it is possible to obtain a relatively short deposition length of the order of 1~2cm with a relatively high deposition efficiency of more than 90%.

The definition of deposition efficiency η is:

$$\eta = \frac{\text{deposition weight of oxides converted from halides}}{\text{total weight of oxides when the halides are completely converted}} \quad (2)$$

The results of the deposition efficiency tests with respect to SiO_2 are shown in Figure 4. The solid line in the figure represents the theoretical value when η = 100%. It was obtained by calculation based on chemical reaction equations.

From the figure one can see that the maximum efficiency η is at around 95%. In general, it is approximately 80%. From these results of experiments which were performed under essentially the same conditions, one can obviously see that: The large Q is (i.e., the more $SiCl_4$ gets consumed) the lower η becomes (as shown by the dotted lines in figure 4). This was mainly due to the fact that the flow rate V_m was excessive. Furthermore, it was discovered that the outlet of the raw material bottle was leaking, which was another reason for the low experimental value of η.

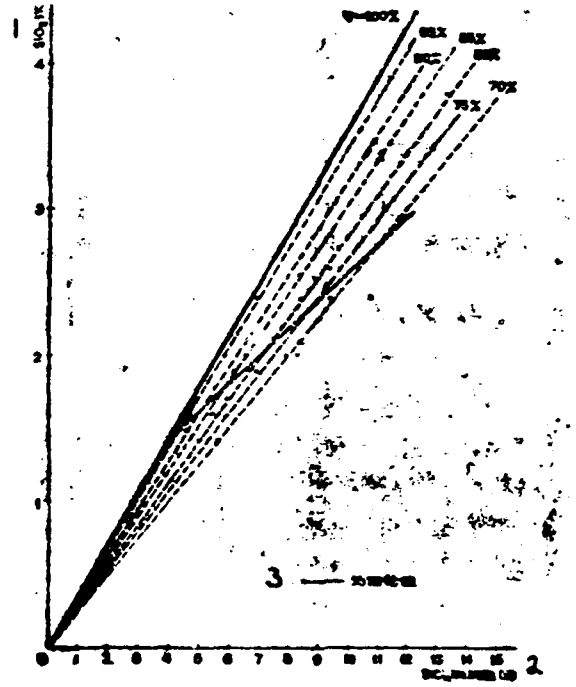

Figure 4. Theoretical and Experimental Values of Deposition Efficiency

Key: 1. Amount of SiO_2 deposited (g)
2. Amount of $SiCl_4$ consumed (g)
3. theoretical

2. Dynamic Experiments

When the resonance cavity was moving back and forth relative to the reactor tube, a homogeneous and transparent thin film was deposited along the inner wall of the reactor tube. It was discovered that when the pre-heating furnace temperature was around 1000°C, despite the fact that the deposition layer was still transparent, the deposition layer showed a large number of bubbles in the contraction stage. When the pre-heating furnace temperature was above 1100°C, such phenomena could be eliminated. According to the analysis made by Küppers et all by neutron activation: the chlorine content in the deposition layer decreases with increasing pre-heating furnace temperature. For example, at 980°C, the chlorine content decreases as the temperature of the pre-heating furnace rises. For instance, at 980°C, the chlorine content was increased to 1 wt%, and at 1050°C the chlorine content was only 0.1 wt%[4]. The major cause of bubble formation is the release of chlorine at high temperatures.

The results obtained in experiments involving doping pure SiO_2 (with GeO_2, P_2O_5, B_2O_3) were satisfactory.

When GE was added, an experiment was carried out to determine the correlation between the index of refraction of SiO_2 glass and the flow rate of the carrier gas. If the dopant concentration is /37 low, then it can be considered that the variation of the index of refraction due to the dopant is proportional to the concentration of the dopant.[5]

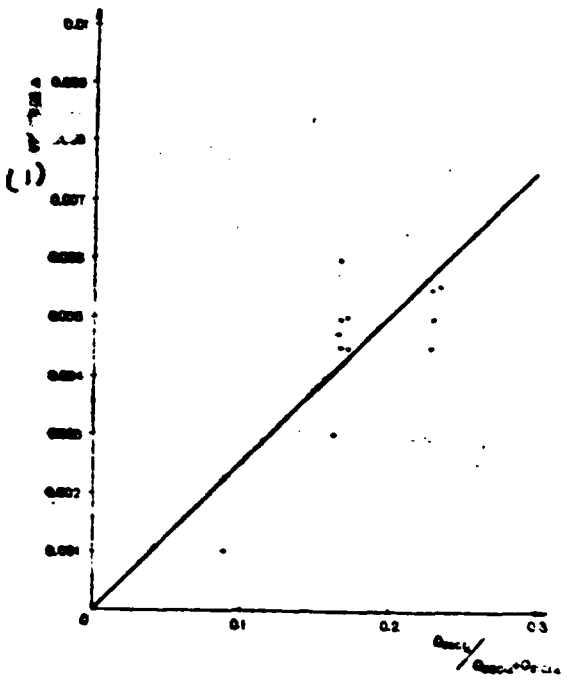

Figure 5. Correlation Between the Difference in Index of Refraction and the Ratio of Carrier Gas Flow Rate

Key: 1. Difference in index of refraction Δ

In the figure, "." is an experiment result. They deviate from a straight line mainly because the accuracy of the flowmeter was not sufficiently high and the height of liquid surface in the reactant bottle was not the same.

3. Fabrication of Multimode Step Index Optical Fibers

The PCVD method has its unique advantages in the fabrication of multimode gradient index optical fibers. Due to the fact that the corresponding flow rate control apparatus is not yet available at the present time, a study of the fabrication of steo index optical fibers was carried out instead.

The optical fiber adopted the structure in Figure 6.

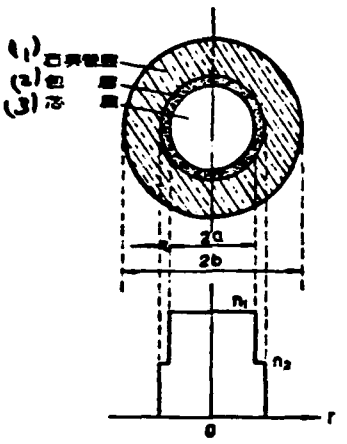

Figure 6. The structure of the optical fiber: outer diameter is 2b; core diameter is 2a, index of refraction is n_1; envelop thickness is t, index of refraction is n_2.

$$\Delta = \frac{n_1 - n_2}{n_1}$$

Key: 1. quartz tube layer
2. envelope layer
3. core layer

In the structure, t is the envelope layer, which is formed by depositing high purity glass. Its functions are to reduce the added loss due to the permeation field in the core and to stop the thermal diffusion of the transition metal ions and OH^- ions in the quartz tube towards the core, in order to improve the loss characteristics of the optical fiber. By summarizing various considerations, it can be determined that the structure of the optical fiber is approximately:

When $\lambda = 0.85\,\mu m$, $\Delta = 1 \sim 0.7\%$, $2b = 125\,\mu m$, $2a = 50\,\mu m$, $t = 10 \sim 15\,\mu m$.

Envelope material: pure SiO_2 glass structure

Core layer material: SiO_2 - GeO_2 binary glass structure

Raw materials: O_2 vs electronic grade. The purity of $SiCl_4$ and $GeCl_4$ should be 99.9999%. Quartz reactor tubes are second grade electrically melted tubes.

The typical data of deposition is: Q_{O_2} =10~20ml/min, Q_{SiCl_4} = 50ml/min, Q_{GeCl_4} = 15ml/min, working pressure P = 8~10 torr, pre-heating furnace temperature T_f = 1150°C, microwave input power P = 150W, cavity traveling speed V = 2.4m/min, traveling distance L = 20cm, approximately 700 deposition envelope layers, and approximately 1000 deposition core layers.

After deposition, the tube was melted and drawn into solid rods approximately 7mm in outer diameter and 20cm in length by heating over a hydron-oxygen flame. Finally, it was drawn into 500m long optical fibers by a graphite furnace. /38

A number of optical fibers were prepared using the aforementioned method. The best fiber is analyzed in the following:

The distribution of the index of refraction of the fiber was measured. The outer diameter of the fiber was found to be 125μm. The core diameter was 44~48μm, and N.A = 0.16. The core had a 2% ellipticity. This was caused by the fact that a positive pressure was not applied in the tube to control the contraction in the fusion stage.

The loss at 0.85μm wavelength was measured to be 4.3dB/km using the cutting method. The measured loss spectrum curve is shown in Figure 7. In the 0.8~0.87μm region. The loss was around 5dB/km. The loss at 0.95μm, which is the absorption peak of OH^-, was as high as 54dB/km. This was due to the contamination of approximately more than 50ppm of OH^-. According to our analysis this was mainly due to the imperfection of the gas handling system. If OH^- contamination can be reduced, the loss across the entire wavelength region can be decreased significantly. The minimum loss occurred at 1.07μm, which was 2.84dB/km.

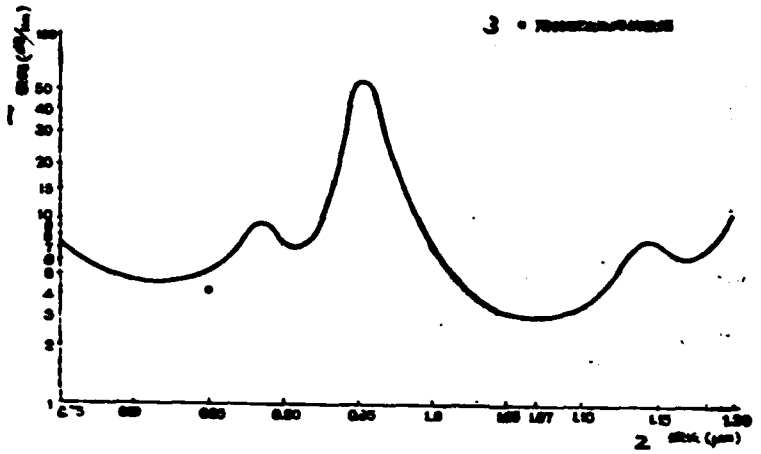

Figure 7. The Loss Spectrum of a GeO_2-SiO_2 Optical Fiber

Key: 1. loss (dB/km)
2. wavelength (μm)
3. data obtained using the cutting method.

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(received on August 10, 1981)

China Scientific Association Held Meeting for Exchange of Working Experience in Editing Academic Periodicals in Beijing.

A meeting to exchange working experience in editing academic periodicals was held in Beijing from November 13-18, 1981.

Over 280 people representing the editing committees and editing departments of various national academic periodicals, the scientific associations of various provinces, cities, and autonomous regions, and organizations in news publishing, distribution and advertising attended the meeting. The key topics discussed in the meeting included: how to combine national economic contruction with operating the periodicals, how to thoroughly execute the "double hundred" policy, how to discover and develop talents, how to fully utilize the function of editing committees by relying on the agressiveness of technical personnel, and how to improve the construction of the editing department itself. Through discussion, the position and effectiveness of academic periodicals were well defined. The experience in running a good periodical was exchanged. Major measures to improve the quality of academic periodicals were studied. Some suggestions were made. Furthermore, the question of establishing an editing workers' association for natural science periodicals in China was raised. The meeting reached the agreement that a planning committee should be formed to establish the association of editing workers. In the meeting, a list of planning committee members was recommended and approved. Furthermore, a planning committee for China Natural Science Periodical Editing Worker Association was officially established.

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(Translation on Page 14.)

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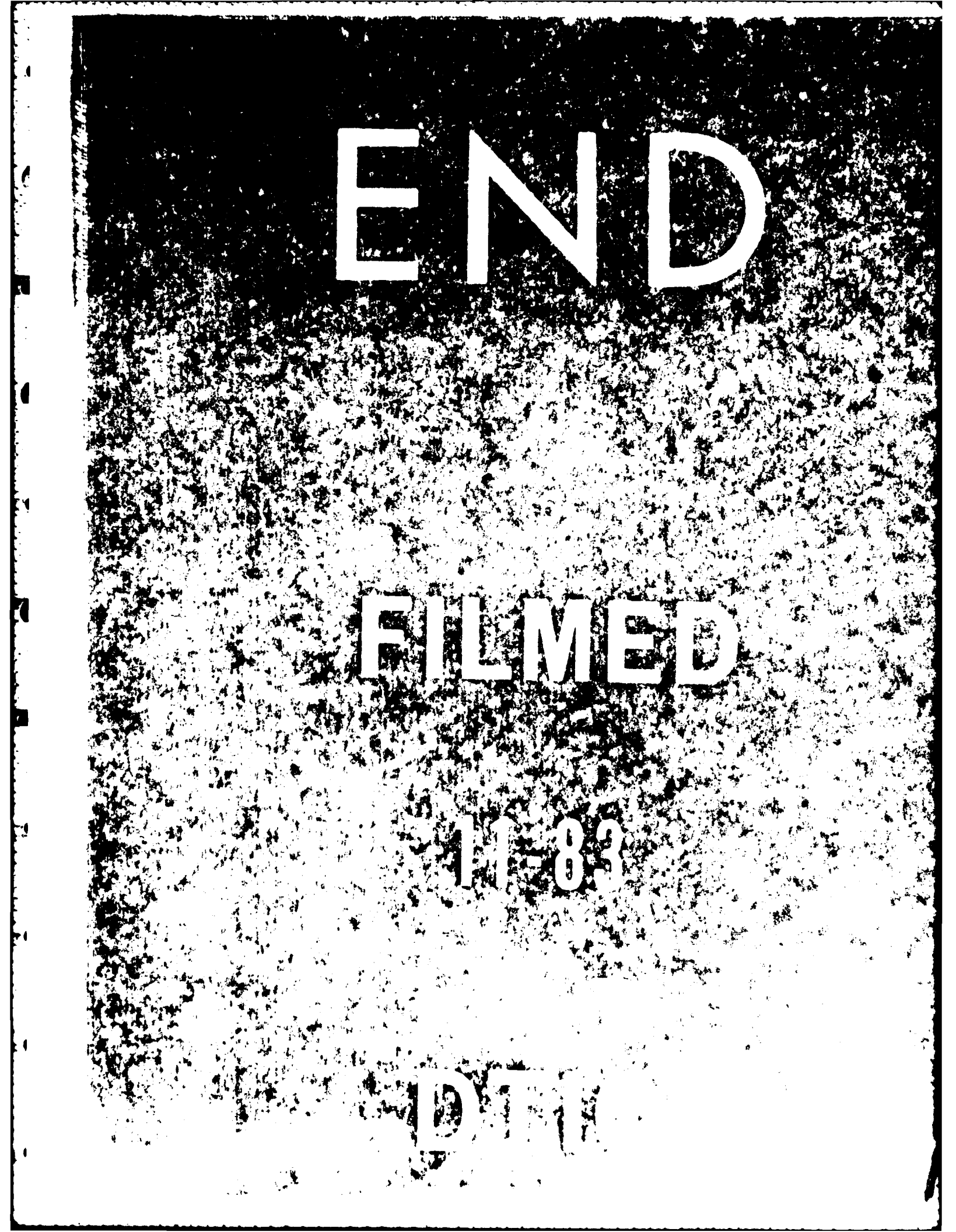
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