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**RESEARCH ON FLAT DISPLAY DEVELOPMENT**

**FINAL REPORT**

**Contract N 91/227**

**Contractor: CENTRE EPITAXY**

**Project N SF/ROK/91/002**

**Trust Fund Donor: GOLDSTAR Co., Ltd., SEOUL, KOREA**

AN ABSTRACT

A research on flat display development is made according to the Contract N 91/227 on the Project SF/ROK/91/002. As initial points for the research are data available in the literature on the devices/technologies related and knowledge/experience of the Contractor on crystallization of thin semiconductor films on amorphous substrates.

In the report followed, first, a brief, critical review of the data available in the literature is given with an emphasis to technologies most acceptable for preparation of poly-Si films on low-melting-point substrates such as Corning Glass 7059.

Second, experiments made by the Contractor, including informations on crystallization apparatus and techniques for several chosen methods, as well as results obtained and their discussions, are described.

Finally, conclusions on the project are given including a brief description of the activity on delivering/evaluation of samples prepared, and a recommendation for future is made to concentrate on one of the method chosen and studied in this research.

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## 1. INTRODUCTION

Flat displays are electronic devices having a broad applications in modern electronics, including computer hardware, high-definition TV, and other consumer related products. Currently these are principal components of the "Lap-Top" computers and small-screen-diagonal TV sets. In a future, as quality of flat displays will be improved and their price will decrease, more and more of the existing, rather cumbersome cathode-ray displays will be replaced by flat ones, and it is predicted that a complete replacement of the devices will occur by end of 90-s. In addition, it is anticipated that, in several years, quality and sizes of the flat displays will reach such a level that high-definition TV based on flat displays will be realized. Other applications of flat displays are also possible.

The perspectives of flat displays are so exciting that many leading electronic companies in USA, Europe, Japan, and other countries engaged in this field. Goldstar Co., Ltd., being one of the international leaders in consumer electronics, is strongly interested in this research.

There are several approaches to realization of flat displays. Of them, that based on liquid-crystal displays (LCD's) seems to be most effective so that such displays become key devices to meet requirements of the different applications.

Performances of the LCD's depend in a decisive manner on quality of silicon films used for preparation of addressing thin film transistor (TFT's) as important components of LCD's. In particular, characteristics of TFT's are principally determined by carrier mobility in the films which, in a turn, depends on microstructure of the films. . Currently, the films used in LCD's are mainly amorphous, and the mobilities are relatively low ( $< 1 \text{ cm}^2/\text{V}\cdot\text{s}$ ) limiting applications of the devices at relatively high frequencies, in color displays, etc.

On the other hand, the mobilities in single-crystalline films can be as high as about  $800 \text{ cm}^2/\text{V}\cdot\text{s}$ . However, single-crystalline

films can be prepared, as a rule, on single-crystalline substrates, which are rather expensive. Moreover, LCD's must be made on transparent substrate, this limits even more a choice of suitable substrates. Usually, various glasses (i.e., amorphous materials) serve as substrates for LCD's. Of known glasses, fused quartz, as relatively high-melting-point material, allows to prepare on it relatively perfect films with rather high mobilities (up to  $500 \text{ cm}^2/\text{V}\cdot\text{s}$ ). However, for many applications, large-area LCD's (e.g., with diagonals as large as about 300 mm) are necessary. Fused-quartz substrates of such sizes would be too expensive for consumer applications. Accordingly, relatively cheap substrates made from, e.g., Corning Glass 7059 are typically used for these applications. Such glasses have rather low-temperature-melting (softening) points. It is, however, well known that, the lower the crystallization temperature, the less qualitative are films grown. Noneless, in view of the high importance of the LCD's, there is a booming interest in devices on low-temperature-melting point glasses. Because permissible working temperatures of such substrates are relatively low, only polycrystalline (rather than single-crystalline) silicon films (poly-Si) can be prepared having carrier mobilities typically  $< 100 \text{ cm}^2/\text{V}\cdot\text{s}$ .

Accordingly, the given project has an aim to optimize conditions for preparation of poly-Si films on low-temperature melting point glasses (such as Corning Glass 7059) for LCD's accenting to lowering temperatures of processes.

In essence, the processes relate to crystal growth, in particular, to nucleation and crystallization/recrystallization phenomena in thin films. Being one of leading organizations in research of thin-film growth, especially on amorphous substrates such as glasses, and responding to a proposal of Korean electronic firm the Goldstar, Co., Ltd. the Centre "EPITAXY" has engaged in the project.

In the following, first, a short review of general ideas of film

growth as a background for our research is given, second, our experiments on preparation of poly-Si films on various substrates and results obtained are described, third, some conclusions with recommendations are given.

## 2. BACKGROUND: GENERAL IDEAS OF FILM GROWTH AS APPLIED TO PRODUCTION OF FLAT PANEL DISPLAYS

If glass wafers (as smooth, plane-parallel plates) are used as substrates for deposition, amorphous or polycrystalline films are typically formed, amorphous ones being formed at relatively low temperatures while, at higher temperatures, the films formed are polycrystalline. Another version is that polycrystalline films are formed from amorphous ones by solid-state transformations. The reason for formation of polycrystalline rather than single-crystalline films is that nucleation of new phases in different areas of the film proceeds independently each of other, hence, the crystalline grains, or crystallites, formed have a broad spectrum of orientations; moreover, they have a broad spectrum of sizes.

The variety of grain orientation and sizes is a principal drawback of polycrystalline films. In the case of semiconductors such as silicon, this variety is especially important because it means a variety of contours and periphery, i.e., a variety of grain boundaries that influences electrical properties of the films, hence, performances of TFT's.

Certainly, the problems with grain boundaries are reduced or even eliminated in single-crystalline or nearly-single-crystalline films. Such films, under some conditions, could be prepared, e.g., on fused-quartz substrates, and this really made by different laboratories and companies.

Below, a short review of a variety of techniques developed during last years for preparation of poly-Si films on glass substrates

with the accent to possibility of their applications for production of active-matrix LCD's is given.

## 2.1. PREPARATION OF POLY-Si FILMS ON GLASS SUBSTRATES

As was noted above, there are at least two approaches to preparation of polycrystalline films. First, to deposit them under conditions which ensure right away formation of crystalline phase. Second, to deposit an amorphous film and, then, to transform it into crystalline one by an additional treatment.

### 2.1.1. PREPARATION OF POLY-Si FILMS BY LPCVD

Poly-Si films are widely produced in microelectronics by low-pressure chemical vapor deposition (LPCVD). Typical deposition conditions are the following: substrate temperatures 600-630°C, pressures in the deposition chamber 0.1-0.5 Torr, flow of monosilane ( $\text{SiH}_4$ ) as a source of silicon [1-14]. As is seen, the deposition temperatures are usually close or slightly exceed the limiting process temperatures of Corning Glass 705° (these are 580-590°C). Sometimes, disilane ( $\text{Si}_2\text{H}_6$ ) or trisilane ( $\text{Si}_3\text{H}_8$ ) are used instead of monosilane for deposition of Si allowing to decrease deposition temperatures down to 420°C (for  $\text{Si}_2\text{H}_6$ ) and 385°C (for  $\text{Si}_3\text{H}_8$ ), however, amorphous (a-Si) rather than poly-Si films are formed. Also a-Si films are formed from  $\text{SiH}_4$  if deposition temperature are lower than 580°C. After deposition, the films are often annealed additionally to improve their structural and electrical properties for TFT. For a-Si deposited from  $\text{SiH}_4$ , an anneal at 550°C for 72 h leads to crystallization, however, no significant grain growth occurs [3]. In the case of using  $\text{Si}_2\text{H}_6$  the annealing is performed at 600-630°C, the best crystallinity is observed in a-Si films prepared at 460-490°C [9,15]. The both noted features can be explained if we take into account interrelations of two principal stages of film formation: nucleation and properly growth.



In the papers [1-14] some general approaches to LPCVD-preparation of poly-Si films were accommodated to the LCD's fabrication. In particular, it was shown that performances of TFT's fabricated in such films can be markedly improved by deposition of the film at much lower pressures (e.g., down to 0.04 and even 0.002 Torr) than normally used in LPCVD [4]. Typically, electron mobilities in the range 10 to 30  $\text{cm}^2/\text{V}\cdot\text{s}$  are reached in such poly-Si films [3,4].

Specially for LCD's a reactor was designed allowing poly-Si films on glass substrates as large as 350x350 mm to be prepared [6].

Finally, from the experiments it was concluded that it is important to maximize, rather than minimize, the excess concentration of point defects promoting grain growth and grain realignment into configurations with lower energy and lower electrical activity [7]. This could be important also in experiments with ion-irradiation-induced crystallization of a-Si (see below, section 2.2).

#### 2.1.2. PREPARATION OF POLY-SI FILMS BY LASER

##### RECRYSTALLIZATION

During the last years, recrystallization of a-Si by short-wave lasers allowing to use relatively-low-temperature processes became rather common procedure in LCD technology.

Recrystallization by Ar<sup>+</sup>-laser. Most commonly, Ar<sup>+</sup>-laser-scanning with a high speed along the film is used for such an aim [16-19]. (Although in [19] fused quartz is used as a substrate, the technique in general allows to recrystallize a-Si on low-melting-point glasses without any damage of the substrates).

Various preceding procedures are used:

Because laser beams are rather energetic, recrystallization temperatures could be rather high reaching even the melting point of Si. In order to avoid damaging the glass substrates, buffer layers

(e.g., 100-150 silicon nitride) are often deposited on them [17, 18].

Amorphous Si films could be deposited either by plasma enhanced CVD (PECVD) at about 300°C [19] or by RF magnetron sputtering at 200°C [18]. In other cases, poly-Si films prepared by LPCVD serve as initial ones for the laser process [16, 17]. In the case of PECVD, a heat treatment at 450°C is important for dehydrogenization of the initial film [19]. No evidence for solid-solid or solid-liquid-solid transformation is given. Only in the paper [19] there is a probability that solid-solid transformation took place resulting in relatively low mobilities ( $50 \text{ cm}^2/\text{V}\cdot\text{s}$ ). In other cases, more probable is zone-melting recrystallization (ZMR) resulting in rather high mobilities (270 to 350 units). In the paper [17], focused elliptical laser beam inclined at 45° to scan direction was used which is known to give nearly single-crystalline films. Owing to relatively high beam velocities (8-20 cm/s), the glass substrates seem to remain undamaged, even at ZMR.

Recrystallization by excimer laser. This short-wave (200-400 nm), short-pulse laser is especially suitable for recrystallization of films on low-melting-point glasses [20-34]. Typical pulse duration is 10 to 30 ns, typical repetition frequencies 10 to 100 Hz, energy densities 100 to 500 mJ/cm<sup>2</sup>. As a result, owing to the high power density, such pulses are able to melt, e.g., 1000-2000 nm Si films on poor-thermal conductive substrates (such as glass). It was estimated that such pulses are absorbed in very thin layers (10 to 20 nm). Further, it was estimated (by calculations) that after melting, such a state remains 100 to 200 ns. After that, during a relatively long time before next pulse, the film is cooled with a sequence of processes or states including supercooling of the melt, nucleation, crystallization from the solid state, etc. Next pulse deals with another system containing different phases. These phenomena were studied both theoretically and experimentally [35, 36] and are very impor-

tant for controlling of film formation.

In practice, a broad laser beam, e.g., 5×5 mm, is used to irradiate PECVD-prepared a-Si films by single or multiple pulses. The films have initial room temperature or preheated up to 500°C, in air or in vacuum. As a result, under pulses with energy densities  $>100$  mJ/cm<sup>2</sup> the films become crystalline with grain sizes in the range 10 to 100 nm. Electron mobilities 10 to 100 units are easily reached, however, inhomogeneties and instabilities inherent in excimer lasers make it difficult to prepare uniform poly-Si films on large areas.

The studies in this direction are continuing.

## 2.2. ION-IMPLANTATION-CONTROLLED THIN FILM CRYSTALLIZATION

Ion implantation, depending on energy and other conditions, can influence strongly on film crystallization processes retarding crystallization (by amorphization of crystalline phase), accelerating it (by intensification of nucleation), improving quality of the films (by facilitation of grain mobility at high density of point defects formed), etc. Accordingly, this procedure combined with other crystal-bearing technologies (various annealing processes, including laser recrystallization) is broadly used in preparation of poly-Si films allowing low-temperature processes [31, 37-49]. Mobilities as high as 100 cm<sup>2</sup>/V-s were reached in the preliminarily ion-implanted films [38, 39]. It was concluded that the influence of the ion bombardment does not come only to film heating, but includes specific ion-solid interaction, and that bulk, i.e., homogeneous rather than heterogeneous (interface) nucleation by Si<sup>+</sup>-implantation is responsible for the crystallization [46].

Many possibilities, still unexplored, gives the approach.

### 2.3. IMPURITY-INDUCED CRYSTALLIZATION

In crystal growth, although not understood completely, influences of impurities on nucleation and on crystallization as general are rather numerous and diverse. Specifically for crystallization of a-Si for LCD applications, impurities such as In, Al, Cu, Ag, Au, P, B, As, Sb influence strongly on kinetics of both nucleation and growth of the crystalline phase of Si decreasing the temperature of the amorphous-to-crystalline transformation and increasing velocity of the growth interface [50-55].

### 2.4. PREPARATION OF NEARLY-SINGLE-CRYSTALLINE SI FILMS ON HIGH-MELTING-POINT SUBSTRATES

Above, it was noted that the higher crystallization temperature, the more perfect films can be prepared. As related to the LCD problem, high-melting-point glass substrates, e.g., fused quartz, are used, and high-temperature processes, such as ZMR, are explored. Although the process was developed generally for integrated circuits and other semiconductor devices [56-64], much attention has been paid to LCD applications [57, 59, 60, 63]. This material (fused quartz) is rather expensive, in the same time, the high-temperature process allows to prepare films with mobilities as high as  $500 \text{ cm}^2/\text{V}\cdot\text{s}$ .

### 2.5. SOME CONCLUSIONS

As is seen, several approaches aimed to preparation of more or less ordered crystalline Si films on glass substrates are developed in connection with LCD, i.e., fabrication of flat-panel displays.

This project aimed to research of preparation of poly-Si films on low-melting-point glasses such as Corning Glass 7059 for flat displays has been performed, first, basing on the data known from the literature, second, using our experience in ZMR and in crystal growth as general.

### 3. EXPERIMENTS ON PREPARATION OF Poly-Si FILMS ON GLASSES. RESULTS AND DISCUSSIONS

#### 3.1. SOME EXPERIMENTAL TECHNIQUES FOR INVESTIGATION OF FILMS

Specificities of objects under consideration are (a) their small thickness,  $< 200$  nm, and (b) polycrystalline nature, i.e., presence of a lot of grains with sizes in a broad range, from nm to  $\mu\text{m}$ . These features make it difficult to control effectively and quickly microstructure of films prepared.

One of the typical criterion of the phase transition from amorphous to crystalline state in thin films on transparent substrate is a change of their optical properties - decrease of absorption and change of reflection in visible spectrum. This was used for fast control of the recrystallization process.

In experiments with ZMR by  $\text{Ar}^+$ -laser recrystallization the film is under heat treatment during a relatively long time ( $\sim 100$   $\mu\text{s}$ ) so that the molten material is able to be redistributed along substrate. In this case, even slight ( $< 5\%$ ) thickness fluctuations are easily detected by optical microscope.

One of drawbacks of PECVD-prepared  $\alpha$ -Si films is rather large (percent range) content of absorbed hydrogen in the films. Rapid evolution of the hydrogen at fast heating of the films results in blistering, hence, in degradation of the films. This phenomenon was fixed by darkening of the films.

Surface roughness of recrystallized films is detected with naked eye (as mat) or by observations in scanning electron microscope.

Microstructure of thin films can be studied, in principle, by a variety of techniques. In our research, most effective was found to be transmission electron microscopy (TEM) of the recrystallized films

"ripped off" the substrates. Most of the photos presented below was obtained in such a way. It is very important that, in addition to micromorphology of the films demonstrating sizes and distribution of crystalline grains, this technique allows to obtain also information about level of crystallinity of the films (from microdiffraction data).

As a complementary technique allowing to estimate average sizes of the grains over relatively large areas electron-diffraction technique was used.

Finally, micromorphology of the films was studied by SEM in secondary-emission mode of observation.

### 3.2. PREPARATION OF INITIAL FILMS FOR RECRYSTALLIZATION

Two principal techniques for preparation of initial films were used in our experiments: LPCVD and PECVD.

The former process was performed at temperatures permissible for the glass substrates (Corning Glass 7059), namely, 580-590°C. At such temperature, very-fine-crystalline films were usually formed. Principal advantage of the film is absence of remarkable amount of dissolved hydrogen negatively influencing on recrystallization process.

The latter process was performed at lower temperature, 250 to 450°C, the higher the temperature, the less is the amounts of dissolved hydrogen, and more perfect are films formed at recrystallization.

Typical thicknesses are 0.1-0.2  $\mu\text{m}$  in the former case, and 0.04-0.2  $\mu\text{m}$  in the latter case.

Also two kinds of buffer were used,  $\text{SiO}_2$  and  $\text{Si}_3\text{N}_4$ , with thicknesses 0.2 to 0.3  $\mu\text{m}$ . The latter is more universal for applications because allows to use both dry and wet processing in fabrication of TFT.

### 3.3. CRYSTALLIZATION OF $\alpha$ -Si BY Ar<sup>+</sup>-LASER

#### 3.3.1. CRYSTALLIZATION APPARATUS

Of different short-wave lasers this one has a feature that it belongs to continuous equipments. Accordingly, principal component of the apparatus is a fastly moving coordinate table.

A scheme of the apparatus is shown in fig.1. An Ar<sup>+</sup>-laser (1) having up to 8 W of multimode radiation with wavelength of 458 and 515 nm. By means of mirror (2) and objective (3) laser beam was focused onto film surface as an elliptic-shaped spot with area about 50x300  $\mu\text{m}^2$ . Sample (4) was installed on the coordinate table (5) fixed vacuum chuck. The table allows to translate the sample in x and y directions coinciding with smaller and larger axes, respectively. The sample could be heated up to 500°C.

#### 3.3.2. CRYSTALLIZATION TECHNIQUE

The table was scanned as follows: it was moved, first, from left to right, then, from right to left along x-axis with a given velocity shifting it in y-direction for a distance  $D \cdot y \cdot d$ , where y is the length of the large beam axis, and  $d < 1$  is the overlapping ratio of two successive passes of the beam. Increasing laser power, some trial experiments were made, from which, by observations in optical microscope, optimal conditions were found. The processes were made in air.

#### 3.3.3. RESULTS AND DISCUSSIONS

Depending on laser power, 3 different crystallization regimes characterized by different morphology and microstructure were observed, two of them are shown in fig.2. The micrographs were obtained by optical microscope in reflected light (fig.2a) and transmitted light (fig.2b). In the figures, relatively light, vertical bands in right side of the photo represent areas where  $\alpha$ -Si were recrystallized via liquid phase, while other areas in the right side were recrystallized via solid phase transformation. The latter areas, as was estimated, contain grains about 50 nm in size (fig.3). Left band (dark in fig.2b) represents non-crystallized area.

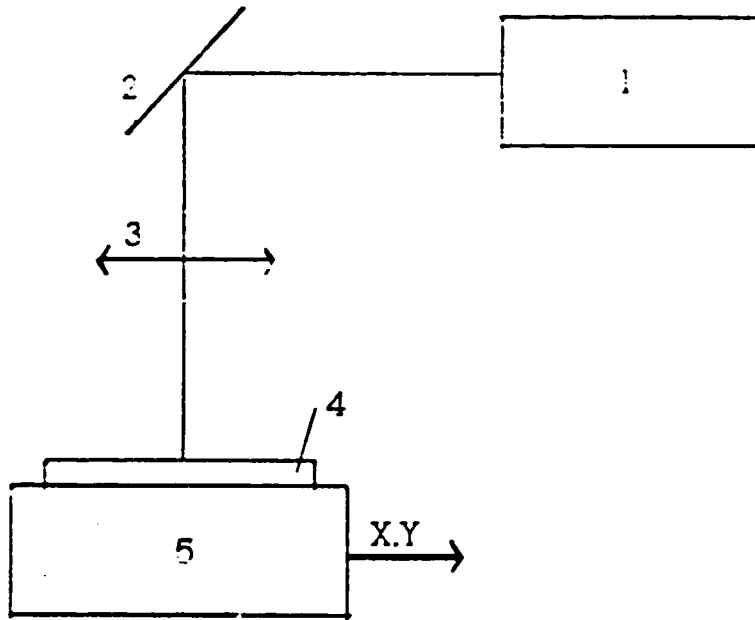
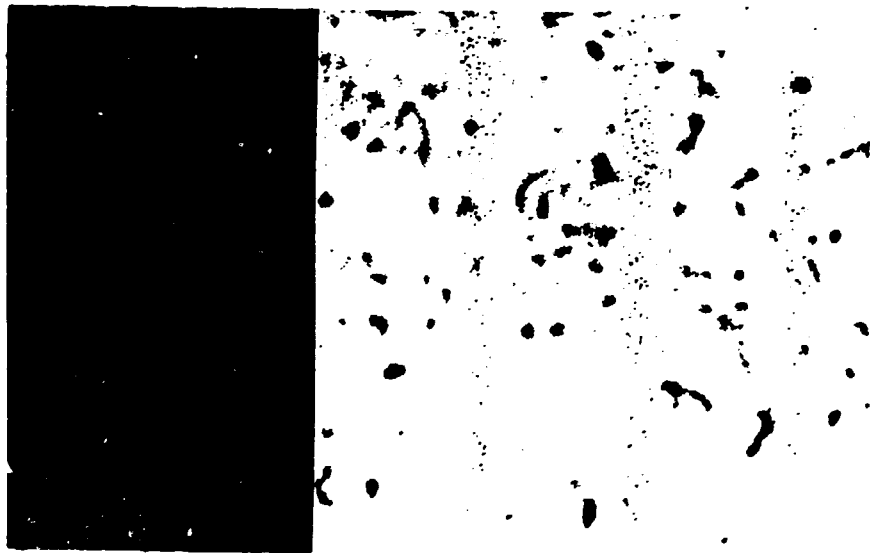


Fig.1. A scheme of an equipment for recrystallization of  $\alpha$ -Si films by  $Ar^+$ -laser.





200  $\mu\text{m}$



200  $\mu\text{m}$

Fig.2. Micromorphology of Si film prepared by  $\text{Ar}^+$  laser recrystallization. Initial films were prepared by LPCVD. Optical micrographs.

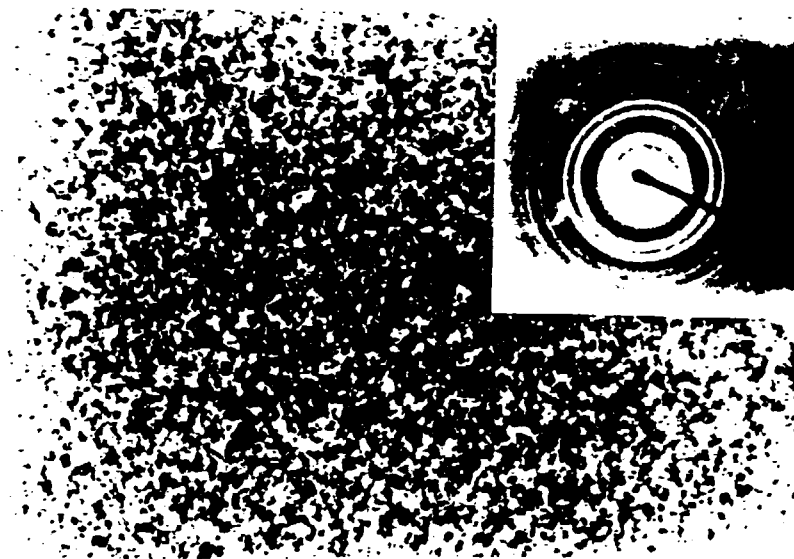


Fig.3. Poly-Si film prepared by Ar<sup>+</sup> laser treatment via solid-phase process. TEM micrograph.

At relatively low powers, the initial  $\alpha$ -Si film melts not completely, but on separate areas forming quasiperiodic structure with alternating liquid and solid phases. As laser power increases, the ratio of the liquid phase increases, while temperature of the film with coexisting two phases is stabilized near the melting point of Si. In such a situation, solidification of each liquid area gives a separate grain. In the same time, the solid areas grow rather quickly forming also some grains. As a result, rather large, up to several  $\mu\text{m}$ , crystalline grains are formed in such a film (fig.4).

Further increase of the laser power melts completely the film, so that a regime of ZMR is realized. Due to a cellular structure of the growth interface, the film formed consists of elongated (in zone-motion direction) crystalline grains, up to 100  $\mu\text{m}$  in length and 5-10  $\mu\text{m}$  in width. The grains can be observed even in optical microscope (fig. 5). TEM study reveals grains which does not contain any defects (fig.6).

In general, the  $\text{Ar}^+$  laser beam technique allows to prepare poly-Si films with rather large grains and even single-crystalline films. However, the technique suffers from some drawbacks.

First, an uniform heating of large (up to 350 mm), fragile glass substrates up to 500°C (as it necessary for the  $\text{Ar}^+$  laser processing) represents a rather difficult problem, in addition, this consumes a lot of time (for heating and cooling).

Second, treatment of multilayered structures by a continuous laser is accompanied by a strong heating of the glass substrate. As a result of a fast heating/cooling cycle rather strong mechanical strains arise in the structures resulting in breaking of the Si film. The strains can be avoided only in island (with sizes about 100  $\mu\text{m}$ ) films.



500 nm

Fig.4. Poly-Si film prepared at intermediate powers. Rather large crystalline grains are seen. TEM micrographs.



50  $\mu\text{m}$

Fig.5. Micromorphology of a poly-Si film prepared by ZMI.  
Elongated grains are seen as vertical striations.  
Optical micrograph.



2  $\mu\text{m}$

Fig.6. TEM micrograph of crystalline grains formed by ZMR,  
at rather large laser powers.

### 3.4. CRYSTALLIZATION OF $\alpha$ -Si BY EXCIMER LASER

#### 3.4.1. CRYSTALLIZATION APPARATUS

A scheme of the apparatus is given in fig.7. As a source of radiation, excimer laser (1) - "LUMONICS-TE-861T" - with Kr:F gas mixture was used giving pulses of 248 nm radiation having durations at half-amplitude (FWHM) about 10 ns with repetition frequencies from 1 to 100 Hz. Pulse energy was about 200 mJ in rectangular frame 22x5 mm<sup>2</sup>. The pulse was attenuated by neutral filters (2). By means of objective (3), slit two-coordinated diaphragm (4) and rotating mirror (5) the beam is projected onto the sample (6) placed on the coordinate table (7). A beam reflected from the objective (3) was directed to the photomultiplier (8), from which a signal was controlled by the oscilloscope (9).

#### 3.4.2. CRYSTALLIZATION TECHNIQUE

By adjusting the system the  $\alpha$ -Si film was recrystallized via melting. The sample was scanned under the beam in *X* direction with velocities from 0.5 to 2 mm/s at repetition rates from 2 to 50 Hz. After each *X* scanning the sample was shifted in *Y* direction for about 90% of laser spot dimension. Initially, the sample has a room temperature. The process was performed in air. Neutral filters were used for stabilization of the laser power and were removed as it decreased due to aging of the gas mixture. Decay of the laser power was controlled by oscilloscope.

As is known from literature [28-30], the higher the power of the laser pulse, the higher is depth of melting and the larger are sizes of grains in the treated film. The maximal laser powers are limited by degradation of the film surface at the some power increasing. For obtaining the optimal conditions of the process we carried out some trial experiments and obtained power threshold of film degradation. Then, we used powers of laser somewhat less than the power threshold.

For excimer-laser technique two different recrystallization approaches were used: recrystallization by a single pulse, and by multiple pulses.

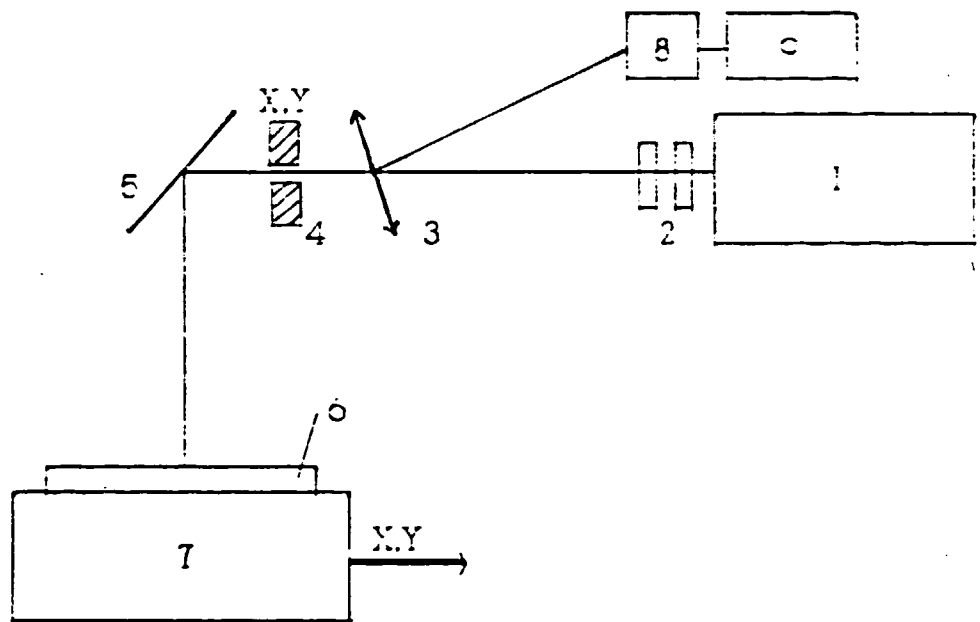


Fig.7. A scheme of an equipment for recrystallization of  $\alpha$ -Si films by excimer laser.

In the first case, the recrystallization process was carried out in X direction by single pulses with overlapping about 10% of the beam width. Uniformity of the energy distribution of the beam was achieved by diaphragming (by Y and X slit diaphragms) so that beam sizes decreased down to about  $1 \times 7 \text{ mm}^2$ . Here, a measurement of the power density was carried out. The total energy was measured by wattmeter placed instead of the sample. Dimensions of beam were determined by measuring of treated area of the single-pulse-treated Si film.

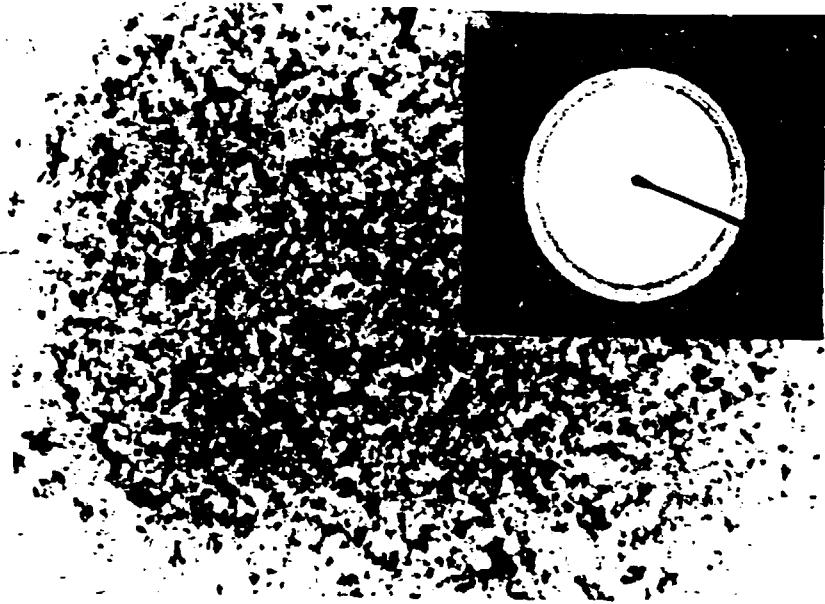
In the second case, according to works of Sony Research Center (by T.Sameshima et al. [20-27]), we used a multipulse recrystallization technique. The beam was diaphragmed to dimensions  $1 \times 7 \text{ mm}^2$ . Along Y axis the beam had relatively uniform energy distribution, however, along X axis it had rather broad gaussian ones. The process parameters (scan velocity and repetition rate) were chosen so that the same points of the film were treated by several pulses with increasing energies. The power of the treatment was determined by the magnitude of energy distribution which were measured using a narrow X diaphragm like described above.

### 3.4.3. RESULTS

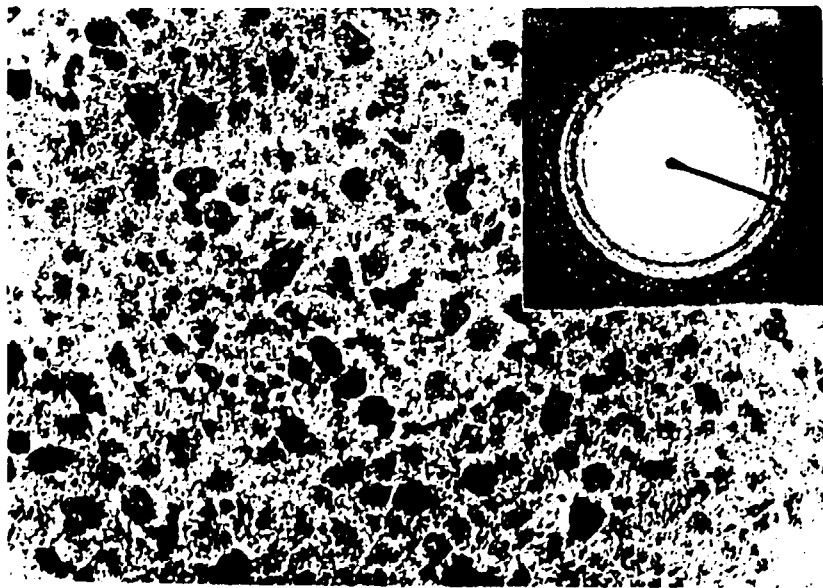
Owing to very short wave lengths of excimer lasers, they allow to treat very thin films, beginning, e.g., 15-20 nm. On the other hand, at treatments of relatively thick films ( $> 200 \text{ nm} = 0.2 \text{ }\mu\text{m}$ ), there is a danger that only subsurface layers will be transformed from amorphous to crystalline state. In figs.8a and 8b are given TEM micrographs of films having different thicknesses, and corresponding microdiffraction pictures. Judging from the figures, it is possible to conclude that, first, in the thinner films grains are, in general, larger (probably, because less nuclei are formed there [27]); second, crystallization in them proceeds more "deeply" (in the microdiffractogram reflexes are more distinct).

Another important parameter of the process is the power density. In fig.9,10 and 11 are given TEM micrographs of 40 nm films prepared at different powers, and corresponding microdiffractograms. As is seen, at re-





a



b

Fig.8. Microstructure of films recrystallized by excimer laser: a - film thickness  $2000\text{\AA}$ ; b -  $400\text{\AA}$ . TEM micrographs together with electron microdiffractograms.

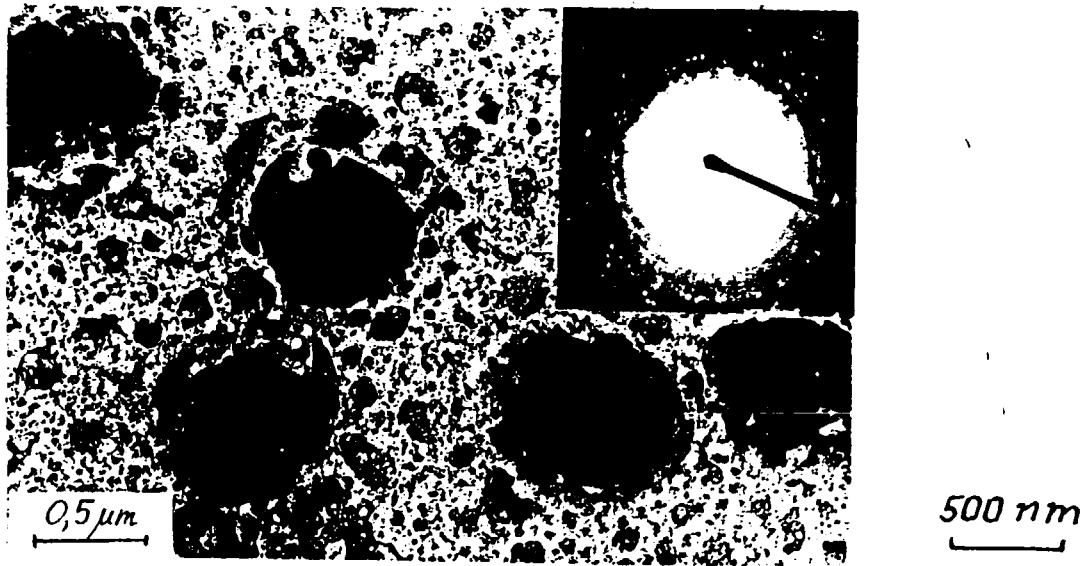


Fig.9. Microstructure of poly-Si film recrystallized at excimer power density  $240 \text{ mJ/cm}^2$ . TEM micrograph and corresponding microdiffractogram.

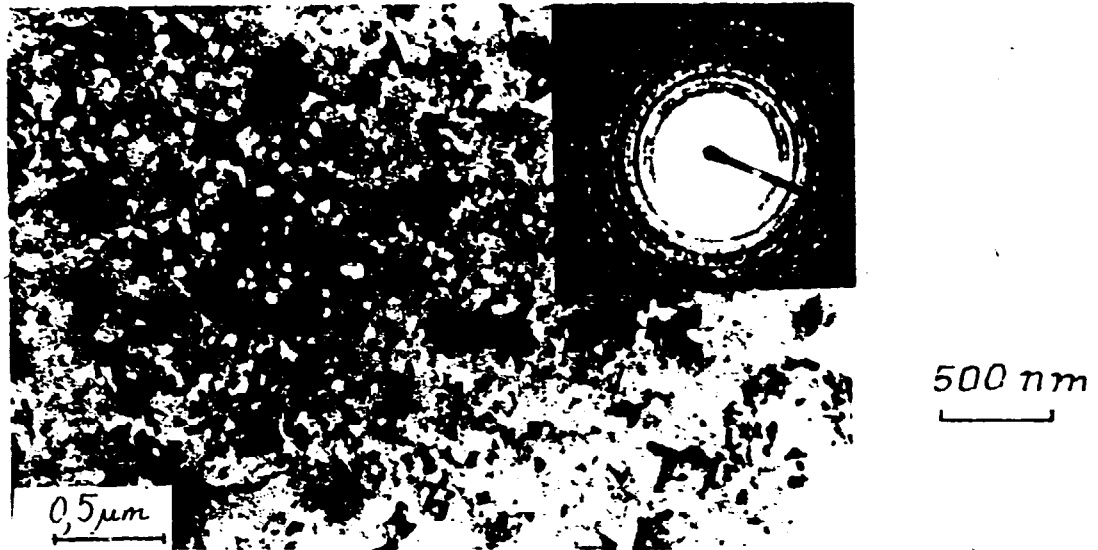


Fig.10. Microstructure of poly-Si film recrystallized at power density  $210 \text{ mJ/cm}^2$ . TEM micrograph and corresponding microdiffractogram.

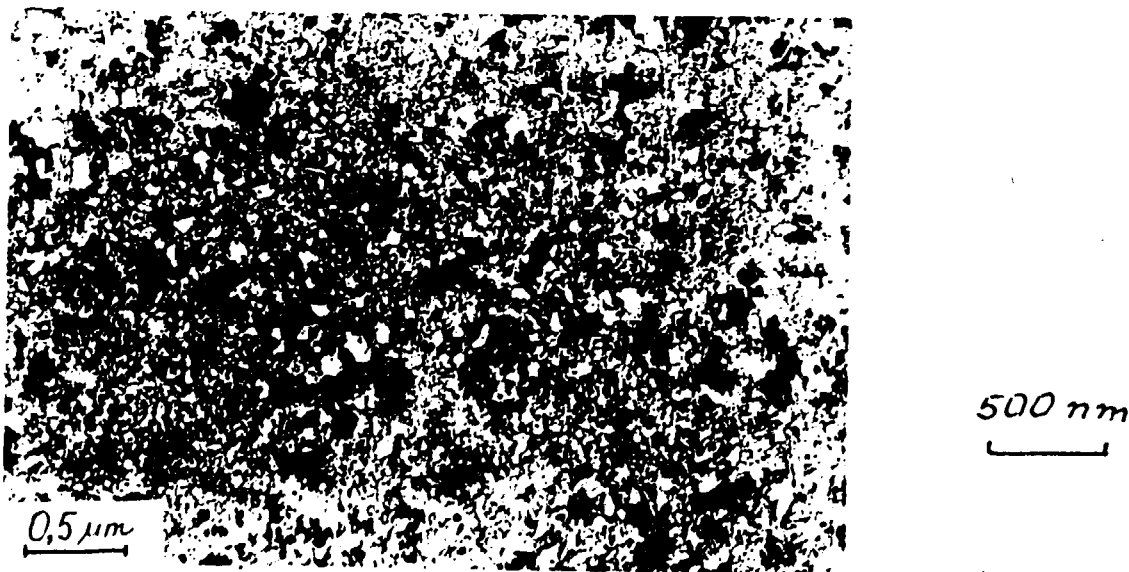


Fig.11. Microstructure of poly-Si film recrystallized at power density  $145 \text{ mJ/cm}^2$ . TEM micrograph.

latively high power (fig.9). some large grains are formed on the background of smaller grains. At smaller power, no such large grains were observed, and the boundary between the two regimes lies somewhere between densities 210 and 240 mJ/cm<sup>2</sup>. At least two features should be noted. First, if compare the microdiffractograms, it is seen that, at lower powers, the grains are more numerous and uniform. Second, if consider the microstructures shown in figs.10 and 11 as preferential for applications, the interval of powers is rather broad (at least from 145 to 210 mJ/cm<sup>2</sup>).

#### 3.4.4. DISCUSSIONS

Best results were obtained for  $\alpha$ -Si films treated by single pulses. In this case, grain sizes reached up to 200 nm. Results for  $\alpha$ -Si:H films are far more poor due to influence of hydrogen. In the second case, first, maximally-accepted power density is smaller (in comparison with  $\alpha$ -Si films), second, non-uniformities appear at repetitive treatments necessary if we desire to have continuous films on large areas.

We suppose that results would be better for thinner  $\alpha$ -Si:H films. In that case, first, total (from surface to substrate) melting of films can be obtained at smaller power densities. Second, it is more easy to remove the dissolved hydrogen. Finally, less probable are simultaneous (competitive) processes of crystallization (and amorphization) from top and bottom surface of the films.

We would like to note some disadvantages of excimer crystallization. First, it is difficult to control power level of excimer laser. Second, the power is continuously decreases as the working gas mixture is aged. Third, the mixture contains halogens that represent some ecological problem in the laboratory room and, especially, for working personnel.

Most simple control of the power (by neutral filters) allows only a discrete (with precision of 8-9%) changes. More smooth control can be made by motion of the objective, and it is this technique which is most suitably for probe experiments. However, this approach is inappropriate for power controlling during fabrication of films because it is accompanied by

changes of the geometry of the laser beam. There are exist some commercial types of excimer lasers with an Intelligent Power Control system which are able to sustain the power at a constant level.

### 3.5. CRYSTALLIZATION OF $\alpha$ -Si BY $\text{Cu}^+$ -LASER

#### 3.5.1. CRYSTALLIZATION APPARATUS

A scheme of crystallization equipment is shown in fig.12. As it seen, the scheme has much of common with that for excimer laser. The  $\text{Cu}^+$  laser (1) had a power 10 W, its radiation has two lines 510 and 580 nm, pulse duration is 20 ns, repetition frequencies about 10 kHz. Inside its resonator formed by mirrors (2) and (3), between the tube (4) and the mirror (3), a rectangular diafragm (5) is placed. A reduced image of the diafragm was projected by means the rotating mirror (6) and objective (7) onto surface of the sample (8). The sample was placed on the coordinate table (9) able to move along X and Y axes and having a vacuum chuck.

#### 3.5.2. CRYSTALLIZATION TECHNIQUE

The sample was fixed on the table by means of the chuck. Adjusting the diafragm, a rectangular beam  $300 \times 30 \mu\text{m}^2$  along the Y and X, respectively, was obtained on the surface of the sample. The sample was scanned under the beam with a velocity in the range 5 to 15 mm/s. The power density was chosen so that the film could be just melted, however, its smooth surface should not be damaged. The recrystallization was performed in air.

#### 3.5.3. RESULTS AND DISCUSSIONS

Under the treatments, surface does not practically changed except of appearing of some weak striations connected with some overlapping (fig.13a). During the pulses (about 20 ns), the substrate is not practically heated to temperatures when any substantial mechanical strains could be developed, hence, microcrackings in recrystallized films were not detected. Judging from TEM research, the films consisted of grains with sizes up to 200 nm (fig.13b).

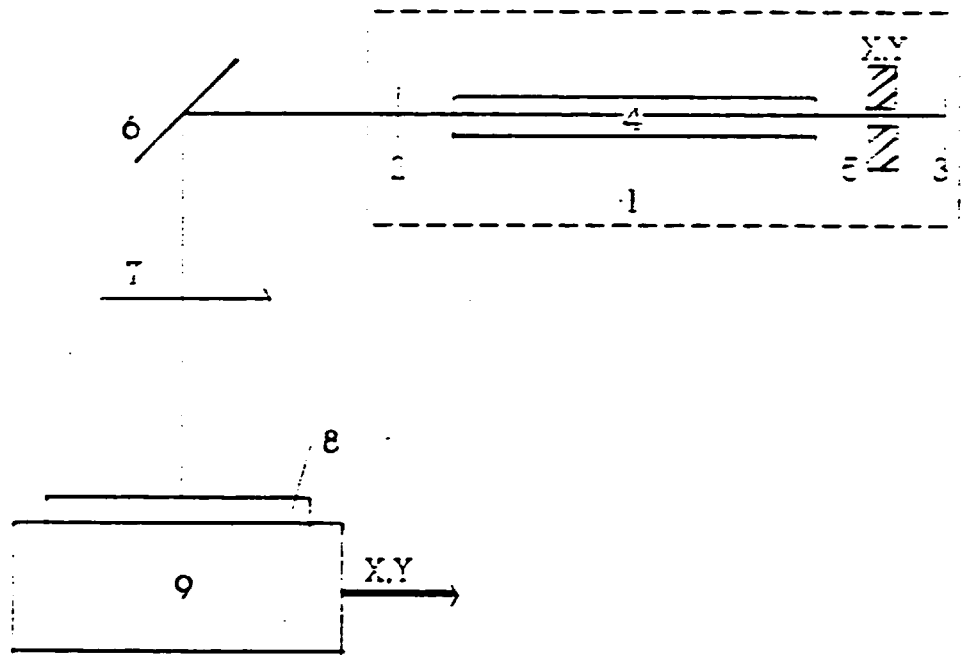
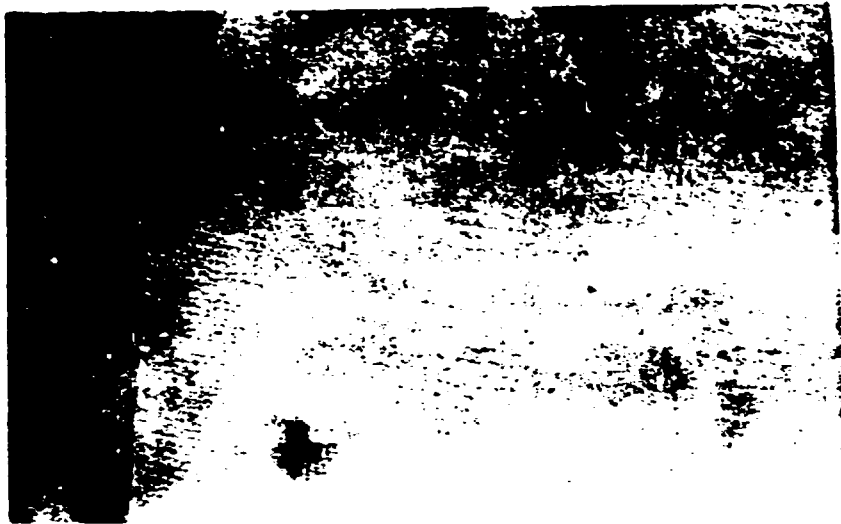
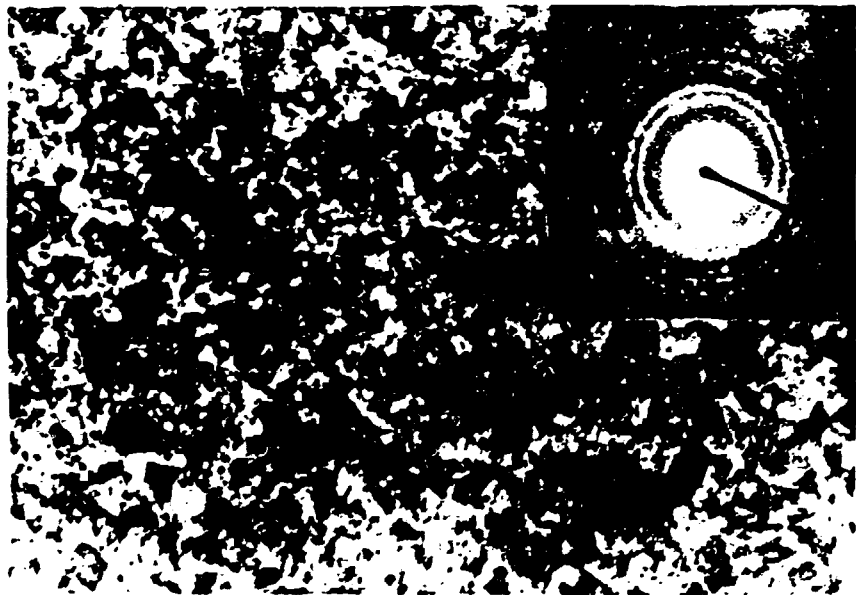


Fig.12. A scheme of an equipment for recrystallization of  $\alpha$ -Si films by  $\text{Cu}^+$  -laser.



a



b

Fig.13. Poly-Si films prepared by  $\text{Cu}^+$  laser recrystallization: a - micromorphology in reflected light of optical microscope: striations are a result of beam overlappings: b - microstructure of the films by TEM and corresponding diffractogram.

### 3.6. CRYSTALLIZATION OF Poly-Si FILMS BY CONTINUOUS YAG:Nd LASER

This technique, in general, is intended for preparation of nearly-single-crystalline films of silicon-on-insulators (SOI) applicable in semiconductor microelectronics for fabrication of CMOS-IS, high-voltage devices, microsensors, etc. [64]. Subboundaries are principal defects in such films. Typically, the process is performed at high temperatures ( $>1200^{\circ}\text{C}$ ), a feature which limits using of this technique for processing with low-melting-point glasses. It is used for preparation of films on fused quartz and on oxidized silicon.

#### 3.6.1. CRYSTALLIZATION APPARATUS

A scheme of an equipment for ZMR process with YAG:Nd laser is given in fig.14. Here, laser (1) gives  $1.06\ \mu\text{m}$  radiation with power about 300 W. By the rotating mirror (2) and cylindrical objective (3) a focused striped beam is formed on a sample placed on the furnace (4) heated by a panel (5) of halogen lamps up to about  $1200^{\circ}\text{C}$ . The furnace can be moved with a velocity about 1 mm/s.

#### 3.6.2. CRYSTALLIZATION TECHNIQUE

A sample (e.g., an oxidized-silicon substrate with the deposited initial poly-Si film) is totally heated by the furnace. By passing the zone through the film it is recrystallized.

In the case of fused quartz, which is difficult to heat by the lamps, a light-absorbing body (e.g., a Si plate) is placed under the sample.

#### 3.6.3. RESULTS AND DISCUSSIONS

The recrystallized film consists of single-crystalline grains elongated in the zone-motion direction (fig.15a). The grains are generally oriented in  $\langle 100 \rangle$  direction, misorientations between them being typically less than  $1^{\circ}$ . Inside the grains no crystallographic defects are usually formed (fig.15b).

In the case of fused-quartz substrates, the microstructure of the film is principally the same as that in fig.15. However, in this case island films should be used in order to avoid cracking due to difference of the thermal expansion coefficients of the films and substrate.



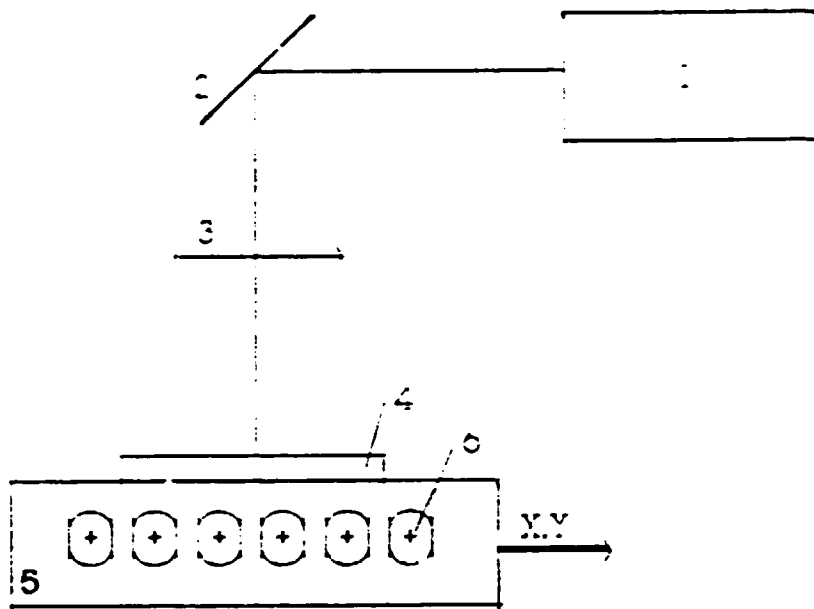


Fig.14. A scheme of an equipment for recrystallization of Si films by YAG:Nd-laser.



a



b

Fig.15. Quasi-single-crystalline Si films on oxidized silicon prepared by ZMR with YAG:Nd laser:  
a - subgrains and subboundaries as revealed by scanning electron microscope; b - microstructure of the films as revealed by TEM.

#### 4. CONCLUSIONS and RECOMMENDATIONS

a) Basing on data available in the literature and on its own knowledge and experience in crystallization of semiconductor films on amorphous substrates, and cooperating closely with the technical staff of Goldstar, the Contractor carried out experiments on preparation of poly-Si films on glass-, fused quartz-, and oxidized silicon substrates.

b) The Contractor evaluated the microstructure and morphology of the prepared films and reported the data obtained to Goldstar.

c) During implementation of the project, in accordance with the Terms of Reference by UNIDO and with the Technical Collaboration Agreement by Goldstar, the Contractor delivered corresponding samples in several parties, totally more 100 pieces, mainly poly-Si films on low-melting-point glasses, to Goldstar.

d) The samples delivered were evaluated by Goldstar as a material applicable for manufacturing TFT's. The data obtained by Goldstar were communicated to the Contractor and used for corrections of the experiments.

e) Basing on the results obtained and taking into account recent news on state-of-art in the flat-display technology, the Contractor recommends to concentrate further efforts in this field on the excimer laser technique.

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