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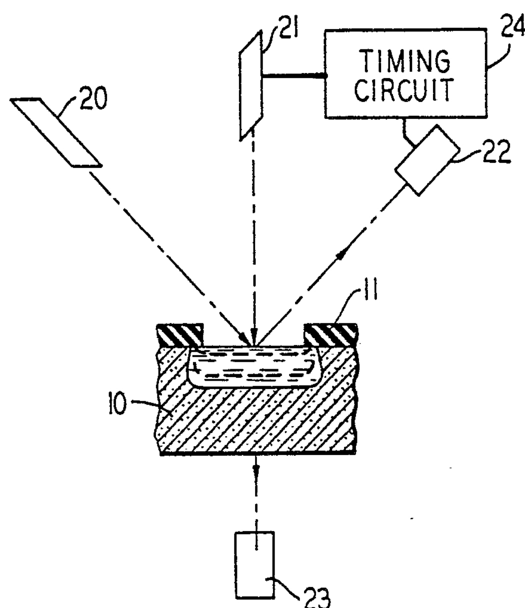

(57) Abstract

For improving the control over a process for annealing surface layers (14) of semiconductor bodies (10) by melting the surface layer by a beam of radiant energy (21), the reflectivity of the surface of the layer is monitored (20, 22) to detect when the surface layer changes its state between molten and solid states, and the amount of radiant energy directed to the surface is controlled (24) in response to such change.

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CONTROL TECHNIQUES FOR ANNEALING SEMICONDUCTORS

Background of the Invention

5 The diffusion of impurities in single crystal semiconductors has been studied thoroughly. The diffusion behavior is well understood and important commercial processes, are based on this understanding.

Recently, considerable experimentation by many 10 investigators has established that short bursts of laser or electron beam radiation can be used to heal radiation damage to semiconductor crystals that occurs during implantation of impurities. See, e.g., G. A. Kachurin and E. V. Nidaev, Sov. Phys, Semiconductors 11, 350 15 (1977); G. Foli, E. Rimini, M. Bertolotti and G. Vitali, Proc. Thin Film Symposium, Atlanta, Georgia, October 1977. (Published by the Electrochemical Society of America, J. E. Baglin and J. M. Poate, ed.); W. L. Brown et al, Proc. Conf. on Rapid Solidification Processing, 20 Reston, VA, November 1977 (Published by the Defense Research Projects Agency and the National Bureau of Standards). In many of those experiments it was found that the boundary of the impurity region moves substantially during the annealing process. The reason 25 for this was not known, since the movement could not be accounted for by solid state diffusion in the short treatment periods involved. Speculation followed, which we have now confirmed, that suitable radiation energy actually melts the regions of the semiconductor on which 30 it is incident and those regions regrow epitaxially. The diffusion of impurities in the molten phase is substantially faster than the solid state rate--fast enough to explain the movement of the boundary.

It is evident then that laser or electron beam 35 annealing is a new processing tool, not only for annealing ion damage effectively in very short time periods, but for relocating impurities in semiconductors on a selective basis. We have devised, in addition, a technique for

precisely controlling the annealing process.

Brief Statement of the Invention

According to the invention, the light reflectivity of the surface of the material is monitored during the annealing process. After the annealing beam is turned on, the surface melts, and the reflectivity of the surface changes. It changes again when the surface solidifies sometime after the beam is turned off. The time difference between these changes is the lifetime of the molten surface. Precise determination of the extent of annealing is obtained in response to the duration of the molten zone lifetime. Control can be effected using this information.

Brief Description of the Drawing

FIG. 1 is a schematic illustration of the impurity redistribution effect occurring during laser annealing;

FIG. 2 is a schematic representation of an apparatus useful for effecting the result described by FIG. 1.

FIG. 3 is a plot showing, as a function of time, t, the intensity of the laser annealing pulse, I_0, the transmitted laser intensity, I_t (in arbitrary units), and the absolute value of the reflectivity R of the surface on which the pulse is incident;

FIG. 4 is a plot of the lifetime of the molten zone as a function of the square of the incident laser energy per unit area E;

FIG. 5 is a plot showing the initial and final impurity distributions for an exemplary laser anneal; and

FIG. 6 is a plot of the final depth of an impurity layer as a function of the measured lifetime of the molten region.

Detailed Description

A qualitative description of the process is aided by FIG. 1 which shows a substrate material 10, which in a preferred embodiment is a semiconductor such as silicon or gallium arsenide, or other material in which

impurities have been implanted. The substrate is covered, but for the window shown, by a masking layer 11 e.g., SiO_2. The laser annealing energy, indicated by the schematic beam 13, is incident on the window. Numeral 12 indicates the initial position of the impurity boundary after (in this embodiment) ion implantation (and in this case before anneal). Upon exposure to the laser beam, the portion illustrated, the boundary of which is denoted by line 14, becomes molten. The impurities diffuse in the liquid phase, forming an ultimate region with a boundary designated 15. In accordance with one aspect of the invention the annealing process can be adapted and controlled either to minimize redistribution or to achieve a controlled movement of impurities. It can be used also to monitor epitaxial regrowth during laser annealing of unimplanted noncrystal material (either amorphous or polycrystalline), e.g., in combination with the formation of silicon deposited sapphire substrates. With reference to the apparatus of FIG. 2 the existence and duration of the molten region is detected by directing a beam from laser 20 or other appropriate radiant energy beam, onto the surface exposed to the beam from the annealing laser 21. A detector 22 for measuring the intensity of the light reflected from that surface is placed so as to receive the reflected beam. A detector 23, situated to receive energy from the annealing laser 21 penetrating the wafer ("transmitted" energy) was also used to obtain the data presented below, but is not necessary for the practice of the invention. A timing circuit 24 is connected as a feedback loop between the detector 22 and the laser 21 as shown. Its use comprises a preferred embodiment in which the extent of laser annealing or the control of the depth of the impurity region is in automatic response to the measured lifetime of the molten region.

The procedure just outlined was demonstrated by the following:

The samples used were 0.25 mm thick (111)

silicon wafers cut from a 1000 Ω-cm p-type dislocation-free floating-zone ingot. They were polished on both faces to permit the observation of the transmitted annealing laser beam. Arsenic was implanted on one face at 50 keV in various doses ranging from 10^{14} to 10^{17} ions/cm^2. Rutherford backscattering spectra showed an impurity profile which peaked at 320 Angstroms from the surface with a 1/e halfwidth of 170 Angstroms.

A Q-switched Nd:glass oscillator and amplifier system was used to generate the annealing beam at a wavelength of 1.06 μm. Mode-control was used in the oscillator to produce a single transverse mode having a Gaussian profile and a diameter (FWHM) of 1.2 mm at the oscillator output and 3.3 mm at the samples. The beam is generated as a series of pulses, the duration of the pulses being 50 nsec (FWHM). With exceptions described hereinafter, the annealing is accomplished by the application of a single pulse to the substrate. By varying the gain of the optical amplifier it was possible to continuously adjust the incident annealing pulse energy from 0.65 J/cm^2 to 16 J/cm^2 and the corresponding peak intensity from 12 MW/cm^2 to 300 MW/cm^2. Both the incident and transmitted 1.06 μm pulses were recorded with high-speed photodiodes and oscilloscopes with response times of 1 nsec. A small (1 mm) aperture was placed behind the sample to ensure that only the central portion of the transmitted beam was measured.

A continuous 0.63 μm HeNe laser was used to measure the optical reflectivity of the implanted surface during the annealing. This beam was focused to a diameter of approximately 0.3 mm and was aligned with the center of the annealing beam at the crystal surface. The angle of incidence of the 0.63 μm beam was 57.5 degrees to the surface normal. A polarizer and quarter wave plate were employed to measure the reflectivity for both perpendicular and parallel polarizations, R_1 and R_{11}, respectively. An avalanche silicon photodiode having a rise time of

approximately 5 nsec was used to detect the reflected beam. Accurate timing was insured by synchronously triggering the recording of the incident and transmitted 1.06 μm pulses and the 0.63 μm reflectivity signal.

5 The extent of the annealing in the samples was determined by Rutherford backscattering and channeling analysis using 1.9 meV alpha particles. For an incident laser energy greater than 1.8 $\pm$ 0.3 J/cm^2 in the 10^{16} ions/cm^2 sample, the channelled spectrum of the annealed sample, had a

10 minimum yield of approximately 3 percent for both the Si and As spectra. This value is comparable to that of unimplanted single-crystal Si. Furthermore it indicates that the complex and extended damage associated with ion implantation has been annealed and that the As has been

15 completely incorporated into substitutional lattice sites. After annealing with the highest energy beam the concentration of As atoms fell to 1/e of its maximum value at a depth of 2000 Angstroms.

The reflectivity measurements are

20 illustrated in FIG. 3. These waveforms have a number of distinguishing features which constitute characteristic "signature" for laser annealing. At time t_o, a laser beam pulse I_o is generated, the pulse, for the particular laser source used (other sources being

25 usable), having the rise and fall time shown. Some of the laser energy penetrates the sample and is illustarted as the transmitted energy pulse I_t. At a time t_1 a sharp transition occurred when the transmitted 1.06 μm energy I_t decreased abruptly, and simultaneously

30 the reflectivity of the monitoring 0.63 μm beam increased from an initial value R^a to a new value R^b. This enhanced reflectivity remained constant until a time t_2 (sometime after the termination of the annealing pulse I_o) when it decreased to a new

35 steady-state value R^c at time t_3. For the 10^{16} ions/cm^2 sample R^a_{11}, R^b_{11}, R^c_{11} were measured to be 0.17, 0.47 and 0.13, respectively, and R^a_1, R^b_1, R^c_1 were 0.63, 0.82 and 0.57. These values

agree to within ± 10 percent with the calculated reflectivities of amorphous silicon, metallic liquid silicon, and crystalline silicon, respectively. The measured values did not change with increasing pulse 5 energy and varied only slightly for different implant doses. Below annealing energies of 1.6 J/cm^2 for the 10^{16} ions/cm^2 sample (2.2 J/cm^2 for 10^{15} ions/cm^2 sample) no appreciable change in reflectivity was observed. As the pulse energy was increased above 10 these values, the duration of the flat portion (between t_1 and t_2) increased reaching a maximum of as much as 800 nsec. This is illustrated in FIG. 4 where the duration of the flat portion of the reflectivity waveform, is plottes as a function of the square of the 15 incident pulse energy per unit area, E. At higher pulse energies, catastrophic surface damage occurred.

The onset of reflectivity change at time t_1 (FIG. 3) was observed to move back towards the onset edge of the incident annealing pulse as the pulse 20 energy increased. The integrated energy of the incident laser pulse from the beginning of the pulse to the time t_1 of reflectivity change was found to be nearly constant for a given sample over the measured range of total pulse energies and found to be 0.9 ± .1 J/cm^2 and 25 1.3 ± .05 J/cm^2 for 10^{16} and 10^{15} ions/cm^2 samples, respectively. The corresponding intensities at t_1 were 34 MW/cm^2 and 49 MW/cm^2 which were also very nearly constant over the measured range of incident pulses.

30 As previously stated, the reflectivities R^b_{11} and R^b_1 are consistent with the measured values of the complex index of refraction of liquid silicon. If the liquid Si were less than 200 Angstroms thick, the observed values of reflectivity at 0.63 μm 35 would differ measurably from those predicted for an infinitely thick liquid. At 1.06 μm the extinction length ($\lambda/4\pi k$) for the transmitted energy through liquid Si is 120 Angstroms which accounts for the abrupt

decrease in transmission of the annealing beam at time t_1 when the surface melts. The constant value of the reflectivity between t_1 and t_2 is strong evidence that the surface remains a liquid during this time
5 interval. After t_1, the absorption of the annealing beam is determined entirely by the optical properties of the liquid, and the beam energy is deposited at a thin layer at the surface.

During the 50 nsec 1.06 μm pulse, heat can only
10 diffuse into the solid a distance of approximately 2 μm and the heat capacity of this volume of hot solid is not sufficient to store all the deposited energy. Most of the incident beam energy deposited at the surface after the time t_1 is stored as latent heat of fusion. For a
15 deposited energy of 1.2 J/cm^2 (corresponding to the longest observed flat-top reflectivity waveform) the liquid layer is approximately 1 μm thick. This distance is longer than the penetration of the As atoms. If we assume the As moves in the liquid by diffusion, then the penetration depth will
20 be approximately $\sqrt{2D_L \tau}$, where τ is the time difference between t_2 and t_1, and the diffusion coefficient of As in liquid silicon is estimated to be $1 \times 10^{-4} cm^2 s^{-1}$. The As will then diffuse approximately 1300 Angstroms. Since the initial distribution was peaked at 320
25 Angstroms, the final distribution would be expected to be 1/e of its maximum experimental value of 2000 Angstroms.

The duration of the "flat-top" portion of the reflectivity signature corresponds to the time required for the complete solidification of the liquid layer at the
30 crystal surface. As illustrated in FIG. 4, this time varied as the square of the incident energy. The time τ is the time required for the absorbed energy per unit area, E_a, to diffuse into the solid so that it is stored in the heat capacity of hot solid (i.e., at $T < T_M$). We estimate
35

$$\tau = \frac{E_a^2}{(T_M - T_o)^2 C^2 D}, \qquad (1)$$

where T_M is the melting temperature (1685 degrees K), T_o is

300 degrees K, C is the specific heat (2.1 J/cm^3 degrees K), and D is the thermal diffusivity in the solid averaged over the temperature profile (0.2 cm^2/sec). Assuming E_a is proportional to E, Equation (1) predicts the
5 observed dependence of τ on E. Taking into account the reflectivity of the liquid surface at 1.06 μm, E_a is 0.23 E. Using Equation (1), we then estimate the slope of the curves in FIG. 2 to be $3 \times 10^{-8} cm^4 sJ^{-2}$. The measured slopes are $1.8 \times 10^{-8} cm^4 sJ^{-2}$ for the $10^{16}/cm^2$ sample and
10 $1.2 \times 10^{-8} cm^4 sJ^{-2}$ for the $10^{15}/cm^2$ sample, in reasonable agreement with the estimate.

In addition to arsenic implanted silicon we have also observed similar reflectivity signatures in silicon (Si), GaAs (Te), GaP (Te and N), InP (Te), as well as
15 crystalline silicon and germanium.

Although these experiments were done in bulk single crystal silicon and an amorphous layer was formed in the silicon as the result of ion implantation of conductivity determining impurities, the
20 layer could also be formed by implantation of "inert" ions such as helium, or argon. Alternatively the layer could be evaporated or otherwise deposited onto a single crystal substrate either of the same material or a material with a matching lattice, thus forming a
25 heterostructure such as Si on sapphire (Al_2O_3). In semiconductor processing it is common to implant impurities into an epitaxially formed layer.

The impurity profiles before and after the annealing process used in one experiment are shown in
30 FIG. 5. The impurities have been redistributed a considerable distance within the crystal.

Also, in this experiment the implanted region was effectively annealed to remove radiation damage at the same time redistribution of the impurities was
35 effected. Alternatively the redistribution could be done as a separate step, or as an error correction or healing measure. Moreover, redistribution of impurities in metals, amorphous materials, or polycrystalline materials can be

achieved with this process without the need for healing radiation damage.

The correlation between the depth to which the impurity region redistributes and the lifetime of the
5 molten zone can be made empirically or can be calculated using liquid-phase diffusion constants. The former are possible using experiments such as that just described. The calculations can follow those just described although the liquid state diffusion constants are not as well
10 established as those for the solid state.

A typical correlation curve based on the data described earlier is shown in FIG. 6. Greatest depth redistribution occurs when the lifetime (t_2-t_1) of the molten zone is longest, thus indicating reflects penetration of
15 the zone to below the initial location of impurities. The redistribution is small where the depth of the molten layer approximates the impurity depth as is the case for short molten zone lifetimes. The rate of solidification is believed to be a straightforward function so the error can
20 be easily determined. When t_2-t_1 is large compared to the threshold time for melting and/or the impurities reside initially near the surface, redistribution will occur nearly proportionately.

If there is a need for the control to be
25 automatic, a feedback arrangement similar to that suggested schematically in FIG. 1 can be used. For precise automatic control it may be desirable to use more than a single laser pulse. This allows a convenient adjustment if the depth is to be varied from sample to sample. The
30 timing circuit 24 integrates the time that the exposed region is molten and terminates the pulses when the prescribed period is complete. Alternatively the laser power can be adjusted. Such "integration" is necessary because, in some instances, discussed further below,
35 resolidification of the molten region occurs after each pulse and prior to the application of the following pulse. The detector 22 is designed in a straightforward manner to convey the timing signal to timing circuit 24

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only when the surface of the exposed region is reflective.

The following examples illustrate some of the ways in which the exposure control provided by the
5 invention can be utilized. All of the examples rely on the measurement of the integrated lifetime of the molten state at the surface of the sample being annealed.

Example 1

In this example the threshold for melting of the
10 surface is first determined by exposing the surface to laser pulses of increasing energy until a change in surface reflectivity is measured, thus indicating the onset of melting. The details of the procedure are given above. The laser exposure is then continued at the
15 threshold level or slightly above until the desired integrated exposure period is obtained, e.g., to obtain the desired annealing and/or the desired impurity profile. Depending on the laser pulse length a single pulse may be adequate, or many pulses may be used to give
20 finer adjustment of the exposure time. This procedure is especially useful when the range between inadequate power to effect annealing, and excessive power that causes catastrophic damage, is narrow. The size of the range, and its position in terms of power level, may vary
25 significantly from sample to sample depending upon the depth and extent of damage, the surface characteristics, and the laser characteristics. It is largely this reason that makes it useful to monitor each sample, rather than exposing all samples with a similar processing history to
30 a predetermined laser pulse.

Example 2

In this example the duration of a single laser pulse, which may alternatively be considered as a continuous laser beam, is controlled in response to changes
35 in the surface reflectivity. The important measurement in this case is the onset of melting, again assumed to be variable from sample to sample. The amount of power necessary to initiate melting is a complex measure of the

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power being absorbed by the sample which in turn is a function of several variables including the amount of damage, the concentration of impurities, the depth of both, and the surface characteristics of the sample. Therefore, 5 a longer delay between the initial exposure to the laser and the onset of melting suggests the need for longer exposure to achieve annealing. It is, then, useful to measure the onset of melting and control the termination of the laser energy in accordance with the delay between the 10 energization of the laser and that measurement. At this point we only know how to interrelate the delay and the necessary exposure time by empirical processes, as are straightforward in the art.

15 In Example 1 it was suggested that the use of many short pulses rather than one or a few pulses may give an added dimension to controlling the integrated exposure period. In principle it would seem that the pulse repetition rate would not matter since the procedure 20 described here measures the integrated lifetime of the molten state at the surface of the sample and it would not matter whether the surface is continuously or intermittently molten. However, in practice we have found that the surface characteristics change after melting and 25 refreezing so that the power required to initiate melting after refreezing may be different from that needed initially. In fact, if the damage anneals significantly during the first pulse, which is likely if melting occurs, then the sample may not absorb sufficiently from 30 subsequent pulses to remelt. Therefore, we envision use of the procedure outlined in Example 2, i.e., controlling the duration of a single pulse, or the use of pulses with repetition rates that do not allow the sample to solidify at the surface. Pulse repetition rates of the 35 order of 10^6 Hz will normally be adequate when processing conventional semiconductor materials. It may be useful to employ a series of pulses to obtain a long molten state lifetime when, for example, the objective is

to redistribute impurities in addition to annealing damage.

Example 3

This example involves an impurity redistribution during laser exposure. The sample initially is implanted with both boron, a p-type impurity, and arsenic, an n-type impurity. The substrate, prior to implantation, has at least a surface layer relatively lightly doped n-type. Exposure of this structure to laser energy by the techniques described previously to give a molten state with a lifetime of 10^{-6} sec moves the boron deeper into the lightly doped n-layer than the slower moving arsenic, thus creating an n-p-n transistor structure with a base width of approximately 0.5 micrometers, and an emitter depth of approximately 1 micrometer.

The detailed description of this invention is given in terms of annealing with laser radiation. Electron beam radiation is equivalent in nearly all respects to laser radiation for the purposes described. In this connection reference is made to U. S. Patent No. 4,082,958 issued April 4, 1978 to A. H. Kirkpatrick. Although this patent suggests avoiding melting the region to be annealed we have shown that melting can be a desirable objective. The power of the electron beam can be adjusted in a straightforward way in order to reach the melting region where the control principles of the invention will be useful.

Claims

1. A process providing improved control of the annealing of a surface layer (14) of a semiconductor body (10) in which said layer is exposed to
5 a beam of radiant energy (21) for a time period sufficient to melt a portion of said layer, characterized by monitoring (20,22) the reflectivity of the surface of said layer, and controlling (24) said time period in response to a change in the reflectivity indicative of a
10 change in state of said layer between solid and molten states.

2. The process of claim 1
CHARACTERIZED IN THAT
the beam is pulsed at different
15 energy levels and the pulses are controlled in response to changes in the reflectivity of the surface.

3. The method of claim 1
CHARACTERIZED IN THAT
the beam is pulsed at a constant
20 power level and the number of pulses to which the layer is exposed is controlled in response to changes in the reflectivity of the surface.

4. The method of claim 1
CHARACTERIZED IN THAT
25 the layer is exposed to a single beam pulse and the duration of the pulse is controlled in response to changes in the reflectivity of the surface.

FIG. 1

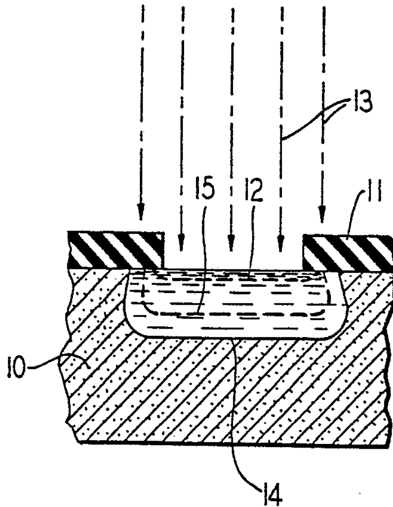


FIG. 2

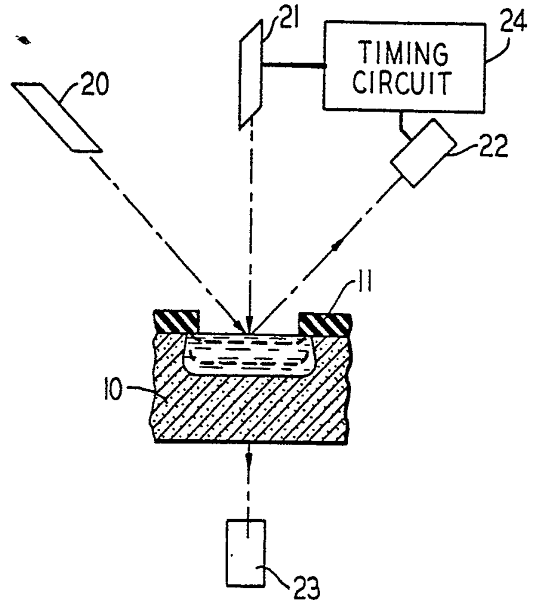
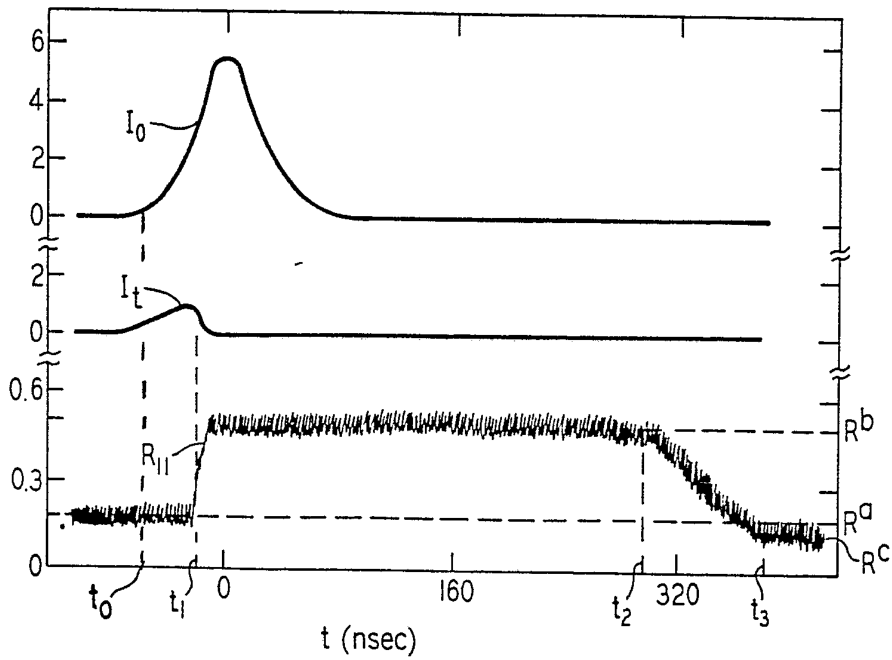


FIG. 3



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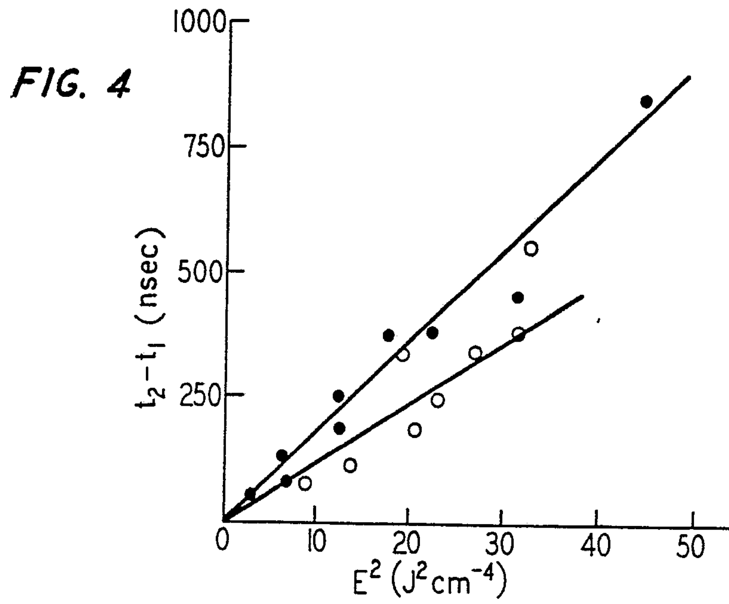

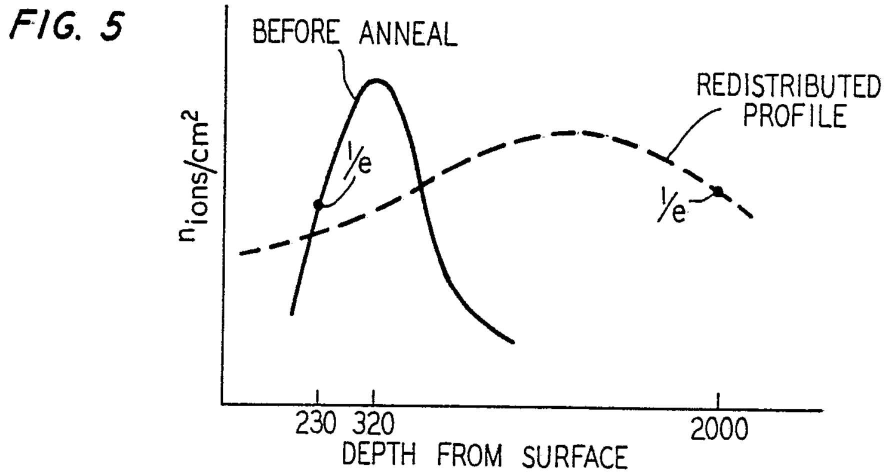

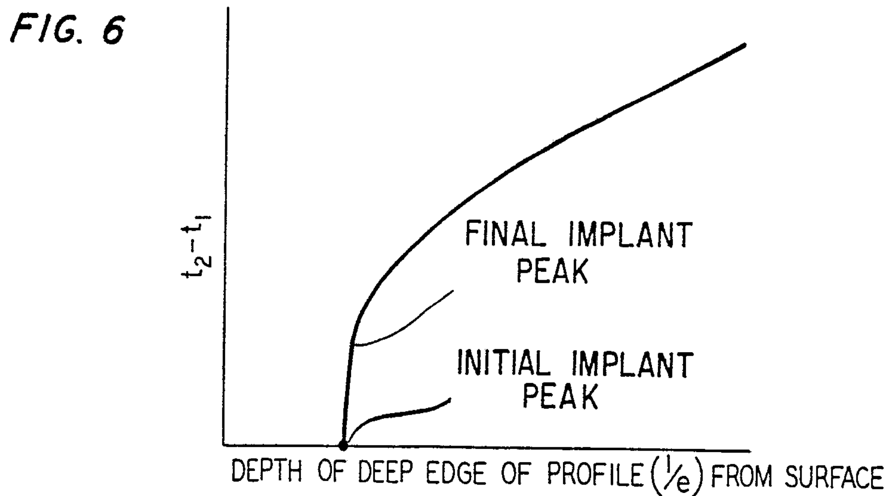

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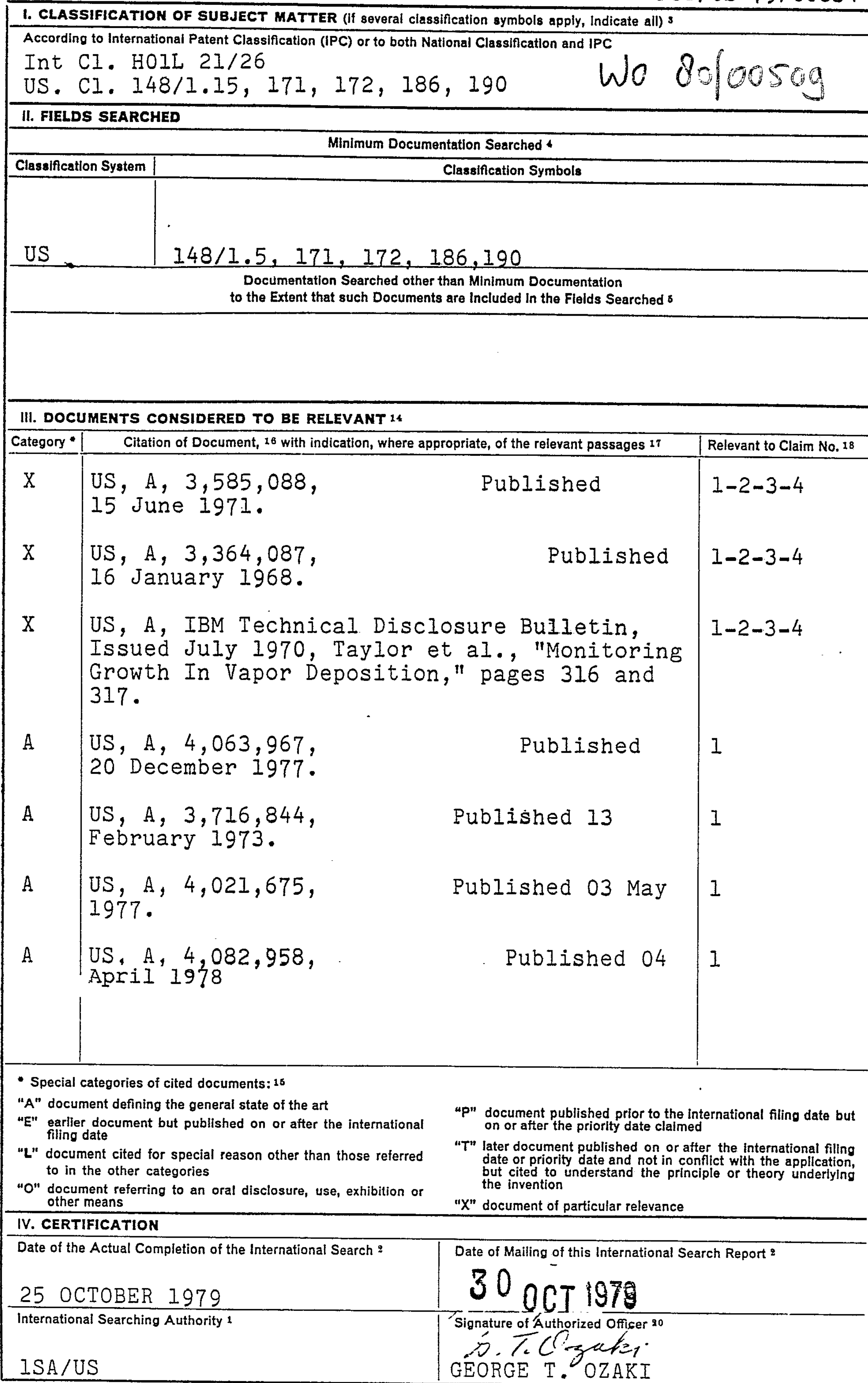

INTERNATIONAL SEARCH REPORT

International Application No PCT/US 79/00834

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [3]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int Cl. H01L 21/26
US. Cl. 148/1.15, 171, 172, 186, 190

WO 80/00509

II. FIELDS SEARCHED

Minimum Documentation Searched [4]

Classification System	Classification Symbols
US	148/1.5, 171, 172, 186,190

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [5]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [14]

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No. [18]
X	US, A, 3,585,088, Published 15 June 1971.	1-2-3-4
X	US, A, 3,364,087, Published 16 January 1968.	1-2-3-4
X	US, A, IBM Technical Disclosure Bulletin, Issued July 1970, Taylor et al., "Monitoring Growth In Vapor Deposition," pages 316 and 317.	1-2-3-4
A	US, A, 4,063,967, Published 20 December 1977.	1
A	US, A, 3,716,844, Published 13 February 1973.	1
A	US, A, 4,021,675, Published 03 May 1977.	1
A	US, A, 4,082,958, Published 04 April 1978	1

* Special categories of cited documents: [15]

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"X" document of particular relevance

IV. CERTIFICATION

Date of the Actual Completion of the International Search [2]	Date of Mailing of this International Search Report [2]
25 OCTOBER 1979	30 OCT 1979
International Searching Authority [1]	**Signature of Authorized Officer [20]**
ISA/US	G. T. Ozaki GEORGE T. OZAKI

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