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Obtaining Target for Solar Cells with Unconventional Supports

The main technological aim is to develop experimental models of magnetron targets of $CuInS_2$ and $CuInSe_2$, controlled Ga doped in concentrations ranging between 7% and 17%. Advantage of using $CuInS_2$ in manufacturing of solar cells is the presence of nontoxic sulfur. The optimum concentration of Ga determine surely the best crystalline phase of $CuInS_2$ and results are an improvement of the absorbtion band and therefore an increase of quantum efficiency of the quaternary mixture – CIGS in double thin layer.

Keywords: *solar cells, unconventional supports, magnetron sputtering*

1. Introduction

The market of electricity to the public use generated by photovoltaic technology based processes increased significantly in recent years [10], [11]. It is true that the energy produced using solar cells is still expensive compared with using conventional methods but is extremely useful for isolated places far from power supply network. The size and power generated by such systems can vary from a few kW to MW. Recent research in the field of "electronics with thin films" focused specifically on the replacement of rigid glass substrates with metal sheet (titanium, stainless steel, molybdenum) or plastic (polyamide, vinyl poliflorure, conductive polymers) are developed [1] - [6].

The advantages of using substrates made of metal or plastic foils are those that, unlike glass, such solar cells are made thinner and lighter than conventional. Also such obtained devices are easy to use and can be used both in stationary applications and the mobile and portable applications (cars, space, mobile phones and even clothes). Problems that may occur are mainly of technological nature but also the compatibility between substrates and semiconductor materials used as energy generators may cause some problems.

The research activities of authors in this field of photovoltaic cells are focused towards the replacement of silicium support by unconventional support of cells. The paper deals with the description of technologies, new methods and experimental results.

In terms of technology, the first problem that can occur is that solar cells are subjected to mechanical work during the manufacturing process that can lead in consequences at their curvature. This has several consequences:

• change the device shape and size

• due to differences in elastic modulus and chemical composition between layers, it can lead to cracking of default layers and less efficient solar cell.

2. Problems due to technological process

Electrical devices and implicitly flexible solar cells are constructed in the form of layers, sometimes by using high temperature processes. Thus, defects can occur due to either mechanical efforts being put on the device, according to Hook's law and, after cooling, due to differences in coefficients of thermal relaxation and moisture of different substrates or films made.

It follows that the determining factors in these cases are: Young's module (Y_s si Y_f) and thickness (d_s si d_f) of the layer (s) and film (f).

When $Y_f \cdot d_f << Y_s \cdot d_s$, the substrate has the dominant role and film makes joint body with the substrate, as solar cells with a rigid substrate. Mechanical effort being put on the substrate in this case is small so that the couple film / substrate curve only very little even if the film is subjected to high mechanical effort. Deformation takes place in two dimensions so that the radius of curvature is:
mechanical effort. Deformation takes place in two dimensions so that the radius of curvature is:

$$R = \frac{d_s}{6\frac{Y_f^*}{Y_s^*}\cdot\frac{d_f}{d_s}\cdot\varepsilon} \cdot \frac{\left[1-\frac{Y_f^*}{Y_s^*}\cdot\left(\frac{d_f}{d_s}\right)^2\right]^2 + 4\frac{Y_f^*}{Y_s^*}\cdot\frac{d_f}{d_s}\cdot\left(1+\frac{d_f}{d_s}\right)^2}{1+\frac{d_f}{d_s}}$$

(1)

Where:

$$Y_f^* = \frac{Y_f}{1-\nu_f} \qquad\qquad Y_s^* = \frac{Y_s}{1-\nu_s}$$

are coefficients of biaxial stretching of film and implicitly of the substrate;

ν_f si ν_s represents Poisson reports for film and substrate;

ε is the coefficient of losses due to deformation. ε has two main components. A component is due to loss of thermal deformation caused by the difference between

20

expansion coefficients of the substrate, a_s si and of the film a_f. The second component of loss factor due to deformation is the internal deformation of the film, ε_{bi}. Result that:

$$\varepsilon = \left(\alpha_f - \alpha_s\right)\cdot \Delta T + \varepsilon_{bi} \qquad (2)$$

Where:

ΔT is the difference between the temperature at which the deposition take place and room temperature. From the above statement it can be concluded that:

 ➢ $R < 0$ when the film is in a state of compression and structure is bending, the film is on the convex part;
 ➢ $R > 0$ when the film is subjected to tension, the film is in the concave part.

When the devices consist of amorphous silicon layer deposited on a rigid glass $Y_f \cdot d_f << Y_s \cdot d_s$ allowing us to neglect the second report of equation (1) and thus simplify Stoney formula as follows, in such cases R is high:

$$R = \frac{d_s}{6\cdot \dfrac{Y_f^*}{Y_s^*}\cdot \dfrac{d_f}{d_s}\cdot \varepsilon} \qquad (3)$$

A film of rigid material and a substrate which makes body with the film, as for example an amorphous silicon film deposited on a polymer film substrate, may have the products between the modulus of elasticity and thickness $Y_f \cdot d_f \approx Y_s \cdot d_s$ Such behavior of the film and substrate give rise to complications from mechanical point of view. Structure is transformed into a cylinder, with a layer of nitrides of silicon with 500 nm thickness, deposited on two different plastic substrates by chemical deposit method in plasma PECVD at 150 ° C. In this case the Stoney formula is no longer valid so that the radius of curvature is:

$$R=\frac{d_s}{6\cdot\dfrac{Y_f}{Y_s}\cdot\dfrac{d_f}{f_s}\cdot\varepsilon}\cdot\left\{\frac{\left[\left[1-\dfrac{Y_f}{Y_s}\cdot\left(\dfrac{d_f}{d_s}\right)^2\right]^2+4\cdot\dfrac{Y_f}{Y_s}\cdot\dfrac{d_f}{d_s}\cdot\left(1+\dfrac{d_f}{d_s}\right)^2\right]\left[\left(1-v_s^2\right)+\left(\dfrac{Y_f}{Y_f}\cdot\dfrac{d_f}{d_s}\right)^2\left(1-v_f^2\right)\right]}{\left(1+\dfrac{d_f}{d_s}\right)\cdot\left(1+\dfrac{Y_f}{Y_s}\cdot\dfrac{d_f}{d_s}\right)\left[\left(1-v_s^2\right)\left(1+v_f\right)+\dfrac{Y_f}{Y_s}\cdot\dfrac{d_f}{d_s}\left(1-v_f^2\right)\left(1+v_s\right)\right]}+\right.$$

$$\left.+\frac{3\cdot\left(\dfrac{Y_f}{Y_s}\cdot\dfrac{d_f}{d_s}\right)^2\left(1+\dfrac{d_f}{d_s}\right)^2\left[\left(1-v_s^2\right)+\left(1-v_f^2\right)\right]+2\cdot\dfrac{Y_f}{Y_s}\cdot\dfrac{d_f}{d_s}\cdot\left(1-v_s v_f\right)\cdot\left(1+\dfrac{Y_f}{Y_s}\cdot\dfrac{d_f}{d_s}\right)\cdot\left(1+\dfrac{Y_f}{Y_s}\cdot\left(\dfrac{d_f}{d_s}\right)^3\right)}{\left(1+\dfrac{d_f}{d_s}\right)\cdot\left(1+\dfrac{Y_f}{Y_s}\cdot\dfrac{d_f}{d_s}\right)\left[\left(1-v_s^2\right)\left(1+v_f\right)+\dfrac{Y_f}{Y_s}\cdot\dfrac{d_f}{d_s}\left(1-v_f^2\right)\left(1+v_f\right)\right]}\right\} \qquad (4)$$

Where:

$$Y_f' = \frac{Y_f}{1 - v_f^2} \quad \text{and} \quad Y_s' = \frac{Y_s}{1 - v_s^2}$$ are coefficients of elongation in the plane of the film deposited and respectively of the substrate.
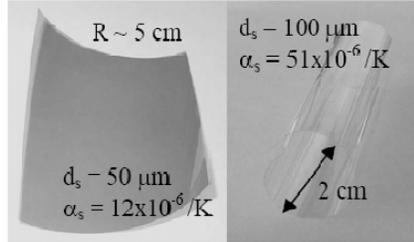
Figure 1. Nitrides of silicon layer thickness 500 nm deposited on two different plastic substrates by chemical deposit method in plasma at 150 ° C

If reports Poisson of v of the film and substrate are identical, equation (4) is simplified as:

$$R = \frac{d_s}{6 \cdot \frac{Y_f'}{Y_s'} \cdot \frac{d_f}{f_s} \cdot \varepsilon} \cdot \frac{\left[1 - \frac{Y_f'}{Y_s'} \cdot \left(\frac{d_f}{d_s}\right)^2\right]^2 + 4 \cdot \frac{Y_f'}{Y_s'} \cdot \frac{d_f}{d_s} \cdot \left(1 + \frac{d_f}{d_s}\right)^2}{\left(1 + v_f\right)\left(1 + \frac{d_f}{d_s}\right)} \tag{5}$$

The rate of loss due to deformation ε is given by equation (2). If the mechanical properties of film and substrate and the radius of curvature R are known, we can calculate internal deformation of the film submitted, ε_{bi}. It is very important to maintain large radius of curvature (flatness conservation areas), preferably even infinite, since this is an important factor in the fabrication of solar cells. Following the analysis equation (5) can easily conclude that if $Y_f \cdot d_f << Y_s \cdot d_s$, R is large.

This can be done as follows:
- choosing a thick substrate (something undesirable for devices that are intended to be flexible);
- keeping the thickness of the entire device very small (there is possibility that this is viable);
- choosing a substrate with a high elastic modulus Young (not for plastic substrates), or combining all these variants.

Another way in which R may also increase if the rate of loss due to deformation ε → 0, namely:
- choosing a substrate with a coefficient of thermal deformation (TD) close to that of film deposited. This is not possible for plastic substrates whose CDT is usu-

ally much higher than the Si film interface. But stainless steel substrate has a relatively low CDT;

- decreasing the deposition temperature of the film we can decrease ΔT. In case of thin film of Si technology deposition, if the deposition temperature drops films obtained have low electrical properties. In this case, affordable electronic devices are the one based on conductive polymers such as LEDs and thin film transistors (TFT); Internal deformation of the deposed film, ε_{bi} depends on the conditions of deposit. There are two other additional solutions to correct unwanted deformations, namely:

- Attach the foil substrate to another rigid substrate ("carrier") to increase device reliability. This rigid substrate attenuates deformation of the structure and has just one tension of distortion in the plane due to small thermal deformation of the film due to "carrier". This approach allows use several types of plastic substrates. However, in this case, we need an adhesive whose temperature of vitreous transition (Tg) and decomposition is higher than the highest temperature at which the process take place. In addition, the adhesive should not be purified with vacuum and has to resist both moisture and dry air. Once the manufacturing process is completed, the adhesive should allow the separation of "carrier" by the foil substrate. Even if this solution was demonstrated in the laboratory, there is not yet an industrial method of manufacturing such solar cells.

- The second solution is the deposition of layers in the form of "islands", so that the deformation force is concentrated around the sides of "islands".

Another problem is the deviation from parallelism (alignment) caused by bending. In Figure 2 are some typical examples of films used in the manufacture of transistors with thin film (TFT) and which are valid for solar cells, made of a thin film of amorphous silicon deposited on a substrate of 50 µm of Kapton. Different types of materials present different types of internal deformation of the deposited film ε_{bi} leading to obtain the different radius of curvature R.
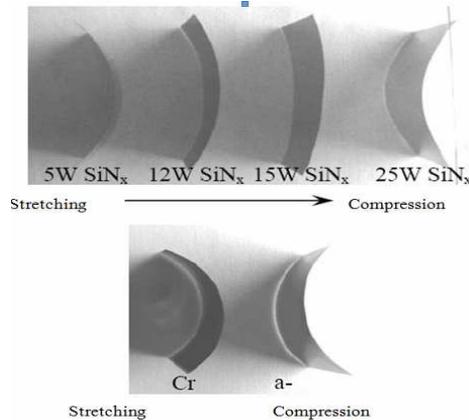
Figure 2. Typical films used in the manufacture of thin film transistors (TFT)

23

It was shown experimentally that a curvature of the device affects the alignment of different layers of the electronic device considered (solar cell, transistor or diode). If the offense starts with the substrate, then it gradually increases as the radius of curvature decreases due to the fact that it creates a higher tension to the device over if it is flat. The higher the radius of curvature is greater, the more deviation from parallelism is lower. For example, with a thin film transistor (TFT) with an amorphous silicon thin film deposited on a substrate of Kapton E, deviation from parallelism between the gate and electrodes source / drain can reach a value of 500 ppm. If an optimized transistor, the deviation can be reduced to ~ 100 ppm. After the set it can be concluded that the radius of curvature of flexible electronic devices must be carefully controlled during the manufacturing process.

It can be seen as there are three different regimes, namely:

- If a proper elastic deformation corresponding to the Sure Regime, current-voltage characteristic is reversible. Electron mobility decreases linearly along with the compressive strength and increases with the stretching force. Mobility changes are correlated with the enlargement and tip growth slope of semiconductor materials conduction band (Si amorphous) of which is manufactured TFT's. For example, for a compressive force of 1%, the mobility of electrons decreases by 25%.

- If the force being put on the electronic equipment is mostly of "compression", not seen any change to a value of mechanical compression force of up to ~ 2%. If "stretching", cracks occur when the stretching force is ~ 0.3% which marks the transition regime. In the transitional regime, TFT is no longer temporary work but regain functions when power is removed. If the stretching force reaches values of ~ 0.5% breaks become permanent and there is a definite mechanical destruction. Such cracks are formed perpendicular to the direction of stretching force. If the direction of current source/drain and of the deformation force are parallel, then cracks cut the power, as shown in figure 4.
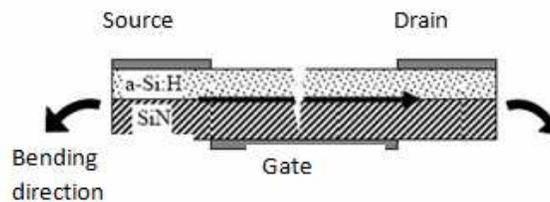
Figure 3. The bending behavior of a TFT

Some studies suggest that the mechanism of destruction of a solar cell-type amorphous silicon germanium is in a certain extent different from that of thin film transistors (TFT) in bending. If the stretching force is ~ 0.7% not notice a change of any parameter characteristic: intensity of short circuit J_{sc}, open circuit voltage V_{oc}, fill factor FF and efficiency η. The same happens if the stretching force is up ~ 1.7%.[12].

24

3. Experiments – make ternary composite targets with controlled GA doping

A. The purpose of the research target technology - processing – deposing photovoltaic element

One of the main aims is the achievement of targets of $CuInS_2$ and $CuInSe_2$ controlled doped with Ga at concentrations between 7% and 17%.

Production of such target materials implicate a delicate process technology and involve the compulsory basic training in few steps:

- Starting materials sorting and convenient choice, in terms of chemical bonds compatibility, of the composite materials and stable of two components. In practical case of targets chosen, they are: Cu - 99.99% pure powder with grain 100 μm-120 μm, In_2S_3 - powder 99.99% pure grit of 114 μm and powder of S - 99.99% pure grain ≤ 100 μm;

- Choice of equipment / furnaces of reaction with automatic control for reaction temperatures (450^0C -1650^0C) and devices for handling and transport of samples;

- Selecting the size of the crucibles of forming of material target, crucible of refractory material and inert to adverse chemical reactions / Al_2O_3 crucible;

- Control and measurement of stoichiometry of target material made in the end process;

- Controlled doping with material for activation - Ga 99.99% pure;

- Develop analysis bulletin by measuring the percentage who achieved doping ternary material.

- Testing the deposits in a thin layer ≤ 1500 nm and physical layer characterization conducted on alternative support (titanium, stainless steel, molybdenum or polyamides, vinyl poliflorure, conductive polymers)[1], [2].

Materials actually used in the preparation of target material have the following characteristics:

Cu

- solid phase
- density - 8.96 g.cm^{-3}
- density in liquid phase - 8.02 g.cm^{-3}
- melting point – 1357.77 K (1084.62°C)
- boiling point - 2835 K (2562°C)
- minerals like: chalcopyrite ($CuFeS_2$), bornite (Cu_5FeS_4), covellite (CuS), chalcocite (Cu_2S) are important sources of copper 99.99% pure copper powder was used in micrometers powder, without using ball mill for uniform grains, but the formation and stabilization of chemical bonds of Cu-S was amplified by using in the melt a mixture of Cu_2S.

Cu_2S

- Class: sulphide
- Color: black/black-blue/blue
- Structure: crystalline solid

25

- Melting point: 1127°C
- Density: 5600 kgm^{-3}

As a third compound important for the chemical balance for stability of the reaction in melting was used In_2S_3 - indium sulphide, as described:

Indium sulphide (In_2S_3)

- Must avoid direct action of: air, moisture, water vapor and acids
- Molecular form - In_2S_3
- Molecular mass - 325.82 g/mol
- Appearance: red powder
- Density in solid form – 4.90 g/cm^3
- Insoluble in water
- Melting point – 1050.0 °C

B. Preliminary laboratory experiments for material synthesis and achieving targets for magnetron sputtering; Optimizing of composite nanostructure with target type CuIn (Ga$^{x\%}$)S$_2$ or CuIn (Ga$^{x\%}$)Se$_2$ and achieve the target for experimental model

Obtaining of uniform films with optimal photovoltaic properties is subjected to the nature of material of target (stoichiometry, degree of homogeneity, chemical stability) and the type of support and not least the conditions of deposit. Preliminary experiments were conducted in laboratory for material synthesis and achieving targets for magnetron sputtering. To optimize the target composite nanostructures was done a review of the latest reporting on obtaining materials $CuInS_2$ simple and doped. Methods of preparation of materials are varied [1]-[9]:

a) Thermal synthesis method from precursors of type SSPs(single source precursors). This methods used microwave radiation to increase the efficiency of preparation of materials. Obtain a uniform material with reduced working time. Were obtained nano particles of chalcopyrite $CuInS_2$.

b) If one starts from the alloy Cu / In films are obtained by sulphating thin films of $CuInS_2$ deposited on glass coated with Mo.

c) Another method is to obtain films of $CuInS_2$ by pyrolysis process. $CuInS_2$ films were obtained by spraying aqueous solution ($CuCl_2$ + $InCl_3$ +$3SCN_2H_4$) on vitreous substrate at temperatures from 250 to 420 ° C. CuInS2 is formed as a product of solid phase reaction of binary sulphides of metals. CuS is formed at 250 ° C by thermal decomposition of complex product $Cu(SCN_2H_4)Cl.1/2$ H_2O. Indium sulphide is formed by the reaction of indium chloride and indium thiocarbamid thermal decomposition.

d) By using alloy targets Cu-In introducing H_2S as working gas and the reaction atmosphere during the deposit by sputtering, films were obtained with S_2 on the glass substrate. Secondary phases Cu-In can coexist when the flow of H_2S is insufficient and excess gas can cause traces of elementary S in the films deposited.

e) CuIn S_2 can be prepared by crystal growth from melt in the ternary system. Cu-InS$_2$-Sb$_2$S$_3$ -Bi$_2$S$_3$.

f) Chemically we can obtain nanowires of CuInS$_2$ with diameter from 30 to 100 nm. Advantage of using CuInS$_2$ to manufacture photovoltaic cells is present of non-toxic sulfur.

g) Optoelectronic properties of CuInS$_2$ depend of chalcopyrite structure defects that can be crystallized in the CuAu structure called vacancies or ionic substitution.

h) Monocrystal of CuInS$_2$ (112) may be obtained by crystal growth from melt in argon atmosphere at pressure ~ 20 bar.

Recent research on phase relations CuS - InS and Cu$_2$S - In$_2$S$_3$ indicate an incongruent melting for CuInS$_2$.

i) By using the hydrothermal method in the presence of polyethylene glycol (PEG). Were obtained CuInS$_2$ and CuInSe$_2$ with different morphologies, spheres, plates, bars and scales.

j) From liquid precursors, the advantage is the reduction of deposition and decomposition temperature to obtain films of CuInS$_2$ or CuIn$_{1-x}$Ga$_x$Se$_2$.

k) Obtain by sequential manufacturing of CuInS$_2$ in first stage involves the submission elements Cu and In on glass support first covered with Mo. Material deposited is heated to 500 ° C with IR light and exposed to the atmosphere of sulfur vapor. Cu and In react with S$_2$ and form CuInS$_2$.

l) Another method is to obtain CuInS$_2$ using three targets. In this case Cu, In and S$_2$ are taken simultaneously as a vapor on a heated substrate. This method is called co-evaporation. It also allows the use of rare earth doping.

For making CuInS$_2$ was used the experience of obtaining materials from the elements or binary compounds. In the first case was proposed to be used Cu, In and S$_2$. Dosing was made at analytical balance, mixing and heat treatment at temperatures 400, 410 and 430 °C. They used aluminium crucibles. Samples were obtained in the form of discs of dimensions 20-30 mm diameter. Samples were obtained marked E1, E2 and E3. The second approach was the method starting from the sulphide of copper and indium. Were applied two types of preparations. Ceramic method of making targets, which consists of preparing on the wet / dry way of a homogenous mixture, pressing mixture followed by heat treatment for sintering. It was used a hand hydraulic press Carl Zeiss Jena Germany with 400Kg/cm^2 maximum pressure. Oven bar superkanthal product type LHT 02/16 made by Nabertherm Germany with maximum temperature of 1660°C and controlled atmosphere of N$_2$. The samples obtained were noted S1, S2 and S3. Starting from binary sulphides have been melting experiments in alumina crucibles at temperatures 1000, 1020 and 1030 °C for 30 min in N$_2$ atmosphere. The samples obtained were noted T1, T2 and T3. Most stable systems in melt solutions were found to be: In$_2$S$_3$-Cu$_2$S-CuS-Cu. This material solidified in the target presented a crystal structure with vacant valence, formed around molecules of In$_2$S$_3$ [2]. Composite material was synthesized of powder with micrometric granulation in the chemical reaction of vapors. Were made quantitative structural analysis by X-ray diffraction,

XRD, and by differential thermal analysis. The oxidation effect in sintering process at temperatures between 980°C - 1045°C was studied by X-ray photoelectron spectroscopy, XPS. After doping with Ga, analytical-quantitative chemical analysis, for highlighting the crystalline structures formed, determined a stability formula formally set on $In_{16}S_{32}$-Cu_v with tetrahedrite and octohedrite formations of In / on mixed valences of Cu and Ga. Ternary target $CuInS_2$ doped with Ga, was made practically using conventional equipment:

-hydraulic press Carl Zeiss Jena / Germany;

-oven superkanthal bar type LHT 02/16 / Germany.

Method of achieving targets were ceramic, the method consists of preparing in the wet environment of a homogenous mixture and pressing followed by sintering.
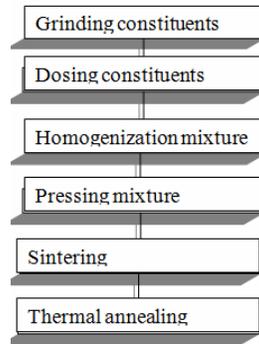
Figure 4. Technological flow scheme

The experiments results are follows:
➢ Sintering material Cu_2S-In_2S_3-CuS : 50g
➢ Material result in the target $CuInS_2$: 39.84 g
➢ Appearance synthesized material: homogeneous and compact
➢ Color: from red to gray-green
➢ Loss of weight:20%

Figure 5. Material $CuInS_2$

28

It can be said as of those shown that during the manufacturing process, devices such as solar cells, field effect transistors and diodes on flexible substrates, meet some problems that need to be resolved. Tension forces that occur in their structure is the result of internal deformation of the film deposited or differences of coefficient of thermal expansion of the film deposited and the substrate, leading to curvature of the electronic device. Following the crookedness, deviations arise at the interface between the layers that form the device. To decrease these deviations is necessary that the manufacturing process to take place with great care to avoid bending.

4. Conclusions

Solar cells of amorphous silicon-germanium type behaves differently from the thin film transistors (TFT) with an amorphous silicon thin film deposited on a flexible substrate, and this difference is greater in expanse. TFT's start a selfdestruction if tension force is $\sim$ 0.3% and destruction is done in a steep. Electrical parameters of solar cell, intensity of short circuit J_{sc}, tension of open circuit V_{oc}, fill factor FF and efficiency η are unchanged till $\sim$ 0,75 and they decrease gradually.

The explanation comes from the fact that the direction of current passing through the device is parallel to the force of stretching and perpendicular to surface cracks appearing on the device, while in case of solar cells is the opposite. Considering the above, and that the growing interest for solar cell application development for mobile equipment (vehicles, artificial satellites, etc.) and portable (computers, cell phones etc.) flexible solar cells represent an ideal solution.

The flexible substrates used are those based on titanium alloys, stainless steel and plastics from polyamides family. These types of substrates are intended to replace conventional Si substrates and glass block used for conventional solar cells. Another type of solar cells are cells that use generally semiconductor nanocrystals type CdTe, CdSe, $CuInSe_2$ (CISe) or $CuInS_2$ (CIS) which deposited on a glass substrate can achieve an efficiency $\geq$ 11%. In future it aims developing of new methods and technologies that allows the use and submission of a polyamide type polymer substrate, given its advantage in terms of cost and weight.

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