

Development of once-through manufacturing machine for large-area Perovskite solar cell production

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ABSTRACT

Perovskite solar cell is a thin-film cell made from solution process including coating, sintering, crystallization, and then encapsulated to become a solar cell that can generate electricity at low cost. At present, the power conversion efficiency of laboratory-produced small cell ($< 1 \text{ cm}^2$) is higher than 25%. Perovskite solar cell is recognized as the next-generation solar cells. However, manufacture technique of large-area Perovskite solar cells is disclosed very few. We developed an innovative low-cost Perovskite solar cell manufacturing machine (MK-20), using once-through process which can continuously carry out thin-film coating and fast heat treatment for four thin layers. We present the design of slot-die coater and fast thermal processor (FTP). The manufacturing process is fully automatic and can make small-quantity sample production with good uniformity and high yield rate. The largest area which can be produced is 80cmx80cm. The maximum production rate is about 6 cells per hour. For a typical p-i-n type Perovskite cell, the trial run on this machine achieves 14.3% power conversion efficiency which is about 77% of small cell produced by spin coating and hot plate annealing. This machine is able to manufacture various kinds of Perovskite cells with different chemical composition having similar fluid properties of Newtonian fluid. Some key technologies in large-area Perovskite cell production are found and also presented.

1. Introduction

Kojima et al (2009) found that the organic inorganic blend of perovskite structure material $\text{CH}_3\text{NH}_3\text{PbI}_3$ which has good sunlight absorbing properties and carrier migration rate can be applied to dye-sensitized solar cells to replace the original small molecule dyes. However, the conversion efficiency is only 3.8% and the liquid electrolyte used will cause corrosion of perovskite material. Kim et al (2012) replaced the liquid electrolyte with a solid-state electron transporting material (Spiro-MeOTAD) and increased conversion efficiency up to 9.7%. This breakthrough began a research boom. The conversion efficiency rapidly increases up to higher than 20% in just five years. The Perovskite solar cell (PSC) can be made with solution process. The cost of solar power generation is expected to be about a quarter of silicon solar cell. Most of researches in PSC focuses on the material science during the past ten years. Although the power conversion efficiency of small-area solar cell (1 cm^2) produced in the laboratory exceeds 23%, there are still many technical barriers to large-scale production that must be overcome. This will begin another boom in the development of perovskite solar cell. Perovskite PV technology

has entered industrialization phase and is beginning to explore the feasibility of various device architectures and manufacturing processes for large area. The upcoming years will be crucial for the future of perovskite PV technology (Nature Energy Editorial, 2020).

The New Energy Center at National Taiwan University in collaboration with E-Sun Precision Industrial Company (Taiwan) has developed an innovative low-cost large-area Perovskite solar cell manufacturing machine using once-through process (called “MK-20”). This machine can continuously carry out four layers thin-film coating and fast heat treatment in sintering and crystallization. The manufacturing process is fully automatic and can make small-quantity sample production. The largest area that can be produced is $80 \text{ cm} \times 80 \text{ cm}$.

2. Manufacturing process of perovskite cell and machine design

2.1. Material of Perovskite cell

The most common structures of Perovskite solar cells (PSC) is composed of several thin layers of organic/inorganic material, including transparent conduction layer (TCO) which is usually made on

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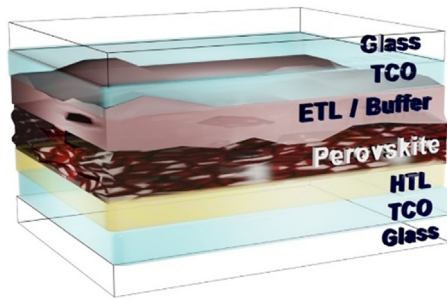


Fig. 1. Four major composition of perovskite cell.

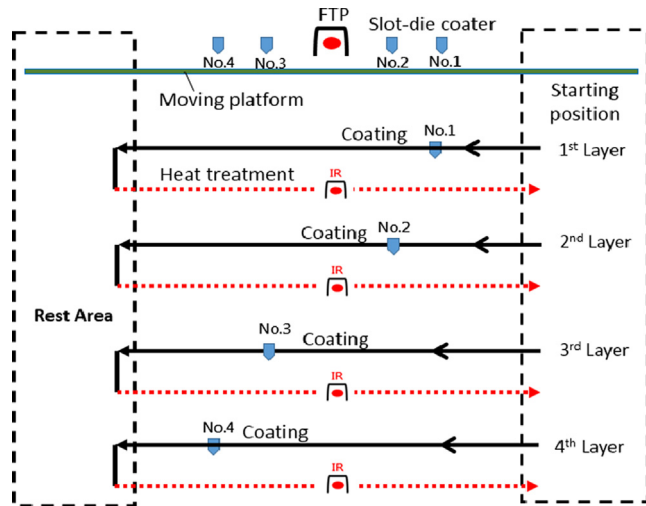


Fig. 2. Schematic of continuous manufacturing process of four layers of thin film in MK-20.

glass as substrate, hole transporting layer (HTL), perovskite layer (PRSK), electron transporting layer (ETL), and buffer/working function layer, back electrode layer (TCO). Fig. 1 shows a typical p-i-n type PSC (Chang et al., 2018). The total thickness of the four essential thin layers (HTL, PRSK, ETL, buffer) is about 500–1000 nm. The manufacture of large-area PSC (> 10 cm × 10 cm) requires challenging technologies which were not found in small-area solar cells (< 1 cm × 1 cm). PSC is a thin film solar cells using solution process which can be manufactured using conventional low-cost chemical process equipment.

2.2 Overall design of manufacturing machine (MK-20)

To reduce the cost and save the space, we designed the manufacturing machine using once-through process (MK-20) which can continuously perform thin-film coating and heat treatment for all four

layers in a single machine. Fig. 2 shows the continuous manufacturing process of four thin layers. The slot-die coating process is adopted. We develop an innovative fast thermal processor using IR lamp (IR FTP) to do the heat treatment. After each film coating, the substrate will be moved to the rest area (left of Fig. 2) for certain period of time for homogenization to obtain a uniform film. Fig. 3 shows the overall configuration of the equipment. The design specification of the machine is shown in Table 1.

2.3. Design of slot-die coater

The slot-die coater in the MK-20 machine is a common coating device to manufacture commercial products such as Scotch tape but requiring a precise control for thin film coating. Fig. 4 shows the schematic of slot-die coating process. Fig. 5 depicts the boundary layer flow of the coated film on the substrate. The coated film thickness is controlled by the solution feeding flow rate Q_d (using a precision syringe pump or similar feeder) and the moving speed of the substrate V_b which draws the solution flow onto the substrate. The volume flow rate Q_f of the coated film over the substrate must equal to the feeding flow rate Q_d and follows Eqs. (1) and (2):

$$Q_f = V_f dL \tag{1}$$

$$Q_d = V_d sL \tag{2}$$

where s is the slot width; L is the flow channel width of slot-die; V_f is the coated film velocity which follows, assuming a shear flow,

$$V_f = (V_b + V_s)/2 \tag{3}$$

where V_b is the substrate speed, and V_s is the free surface velocity of the coated film. Assuming that the film is very thin such that the shear force across the film boundary layer is negligible, i.e. assuming a plug flow, so that $V_s = V_b$. From the mass balance relation $Q_d = Q_f$ or $V_d s = V_b d$, we obtain the theoretical coated film thickness d :

$$d = \frac{V_d s}{V_b} \tag{4}$$

From Eq. (4), the variation of the coated film thickness d with substrate moving speed V_b can be plotted as Fig. 6. It is found theoretically that the coated wet film thickness can be as thin as 1–5 μm or thinner (approaching spin coating result). Fig. 7 shows the operation of the slot-die coater.

2.4. Design of fast thermal processing device

The hole transporting layer (HTL), Perovskite layer (PRSK) and electron transporting layer (ETL) needs heat treatment at different elevated temperature according to the chemical composition. The hole transporting layer (HTL) needs sintering at 200–400 °C and the Perovskite layer needs annealing at around 180–200 °C (Chang et al., 2018). A long tunnel furnace is commonly used in heat treatment but is



Fig. 3. Overall configuration of MK-20.

Table 1
Design specification of MK-20.

<ul style="list-style-type: none"> • Slot-die coater No. of slot-die coaters: 4 Coating speed: 0.2–20 m/min Coated wet film thickness: < 10 μm Max coating width: 800 mm Substrate moving speed: 0.1–3 m/min 	<ul style="list-style-type: none"> • Rapid thermal processor Heater type: IR lamp (changeable) IR heater power: 2–6 kW (adjustable) Surface heating speed: 10–30 K/s Spatial variation of light output intensity: < $\pm 2.5\%$ Moving distance in lighting area: 7 cm
<ul style="list-style-type: none"> • Dimension Overall size: 3000L \times 900D \times 2000H (mm) Working chamber: 2800L \times 1000D \times 1000H (mm) Largest cell area produced: 800 \times 800 (mm) 	<ul style="list-style-type: none"> • Power consumption Power: 220 V/60 Hz Max power: 6 kW • Maximum production rate: 6 cells per hour

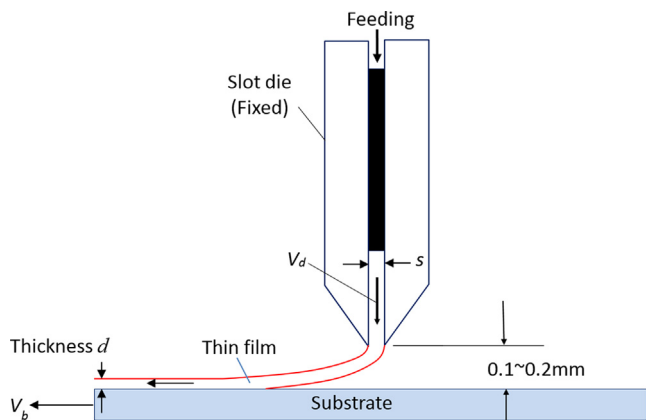


Fig. 4. Schematic of slot-die coating.

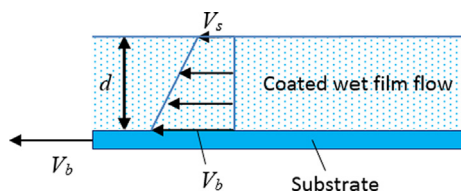


Fig. 5. Schematic of coated wet film flow on substrate.

bulky, high energy consumption, and expensive. We developed a fast thermal processing device (FTP) which is very compact, low energy consumption, and low cost. Fig. 8 shows the schematic of fast thermal process using IR heater. The distance that the substrate moving through the FTP is only 7 cm, just like a commonly-seen scanning device (heat scanner).

The instantaneous surface temperature variation can be estimated using the theory of transient heat conduction in a semi-infinite solid (Holman, 1981). Fig. 9 shows that the surface temperature can be raised 150 °C in 10 s using a high IR radiation intensity about 107 Suns (107 kW/m²) if the absorption coefficient of the solution is greater than 0.4. The spectral absorption coefficient of the solution is an important parameter in thermal processing which should match the spectrum of IR lamp in order to reduce energy consumption. The rate of surface temperature rise higher than 50 K s⁻¹ is achievable if using 6 kW IR heater. The power input of IR heater can be adjusted and fixed by a feedback controller through a SCR. Fig. 10 is the pictures of the rapid thermal processor (IR FTP).

The uniformity of the output light from FTP is quite important. To measure the uniformity of light output, we designed a measuring device which uses a small solar cell as the sensor and mounted on a motor-driven screw guider (Figs. 11 and 12). The sensor is quickly moved through the light output port of FTP. The measured short-circuit current of the solar sensor represents the intensity of light. To reduce the error due to the heating of the sensor by IR heat, the sensor is moved through the entire light output port within 1 s. Fig. 12 shows the measuring device. Fig. 13 shows the variation of relative radiation intensity at the output port. Table 2 shows that the width with light output variation less than $\pm 2.5\%$ is 42.6 cm for 4 kW IR heater.

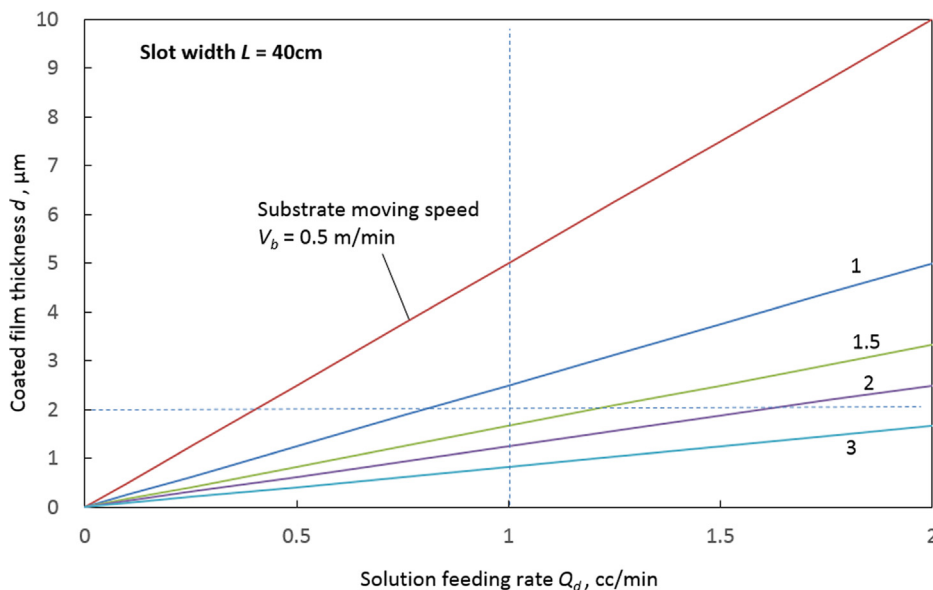


Fig. 6. Variation of coated film thickness.

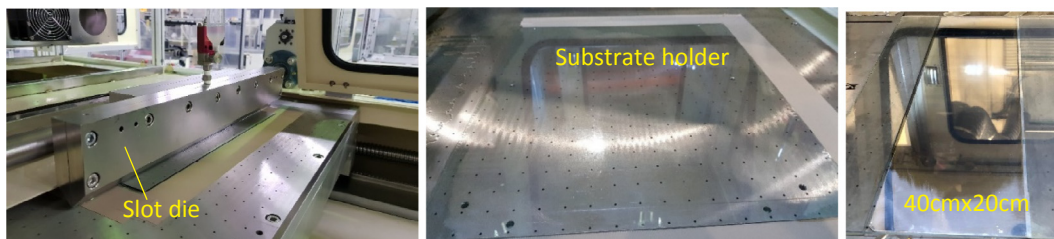


Fig. 7. Operation of slot-die coating.

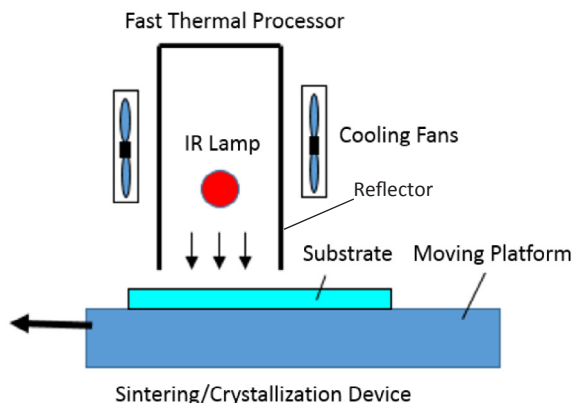


Fig. 8. Schematic of fast thermal process.

The reflector was designed to reflect the IR radiation to create a uniform light output. Four cooling fans were installed to cool the reflector for overheat protection. It will be turned on automatically at high temperature. Fig. 14 shows that no cooling is required for thermal processing time shorter than 40 s. The operation of cooling fan will induce flow disturbance inside the working chamber and affect the manufacturing process of thin films. It is better not using cooling fan during chemical processing. This can be achieved for fast thermal processing.

3. Machine performance

3.1. Automatic operation

This machine can successively carry out coating and heat treatment. The process is completely automatic and it takes about 8 min to complete the four layers of coating and heat treatment as shown in Fig. 15. The maximum production rate is around 6 cells per hour.

3.2. Results

The machine can produce Perovskite cell with maximum dimension 80 cm × 80 cm. We used the p-i-n perovskite cell in the performance test of the machine MK-20. The p-i-n perovskite cell consists of solutions of NiO_xNP_s (HTL), MAPbI₃, PCBM, TBAOH-SnO₂N_s(ETL) (Huang et al., 2020; Chang et al., 2018) which are successively coated on the FTO glass. The thermal annealing (crystallization) of Perovskite layer (MAPbI₃) takes 3–6 s using 4–6 kW IR heater and the sintering of HTL hole-transporting layer (NiO_xNP_s) takes about 20–40 s using 4 kW IR heater.

Fig. 16 shows the coating and heat treatment process for 40 cm × 20 cm cell. Fig. 17 shows the results of coating and sintering of the first layer on the FTO substrate. Fig. 18 shows the result of coating and crystallization of the second layer (MAPbI₃) on NiO_x. Fig. 19 shows the finished Perovskite cells with all 4 layers coated. The uniformity of the products is satisfactory and the yield rate in small quantity production is larger than 80%. Fig. 20 shows the solar power conversion efficiency of produced perovskite cells produced (12 cm × 12 cm). Fig. 21 shows the measured I-V curve of perovskite cell ever produced. The power conversion efficiency (PCE) is 14.3% which is about 77% of small cell produced by spin coating and hot plate annealing (Huang

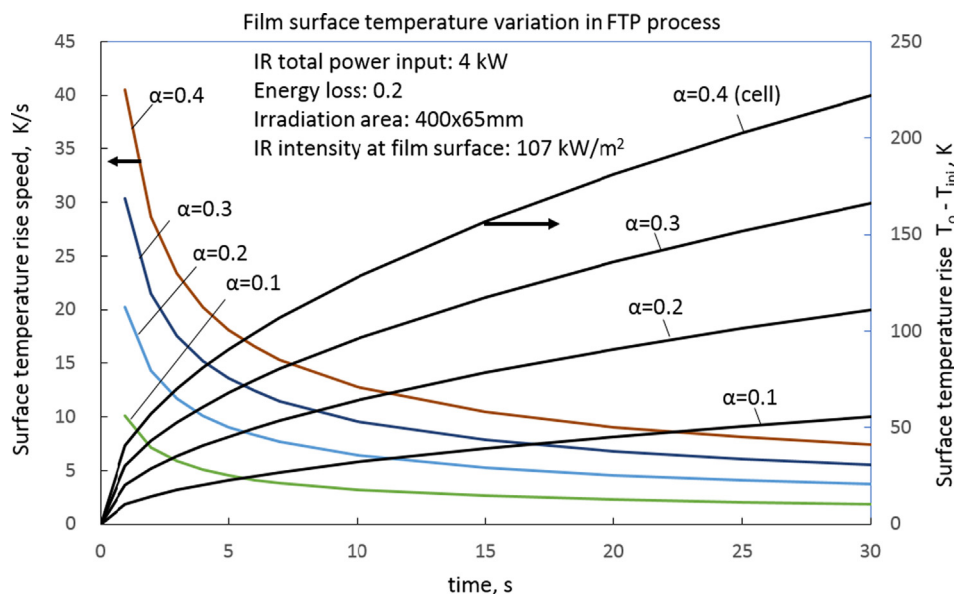


Fig. 9. Instantaneous surface temperature rise.

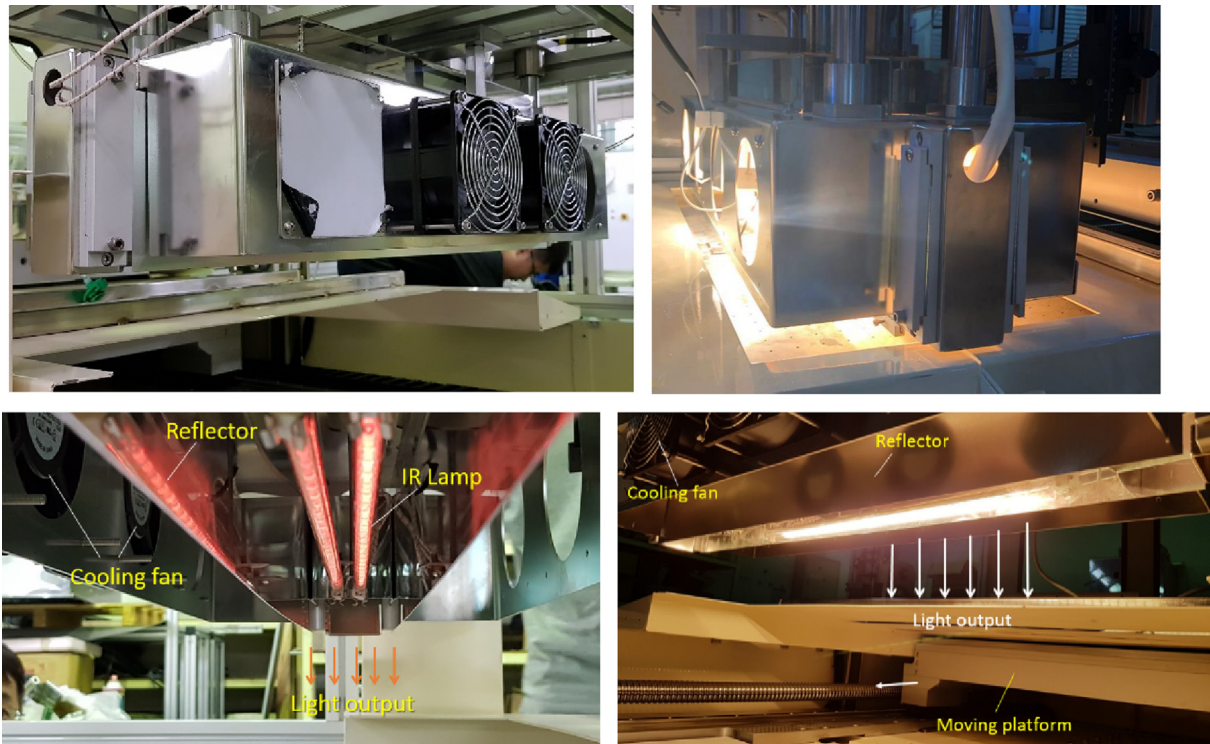


Fig. 10. The fast thermal processor (FTP).

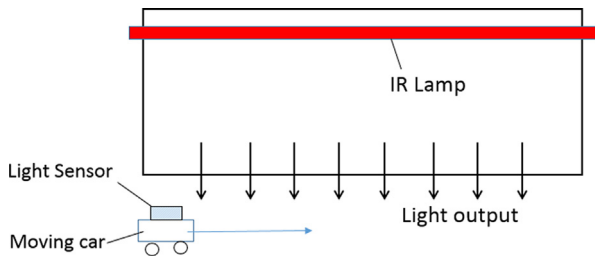


Fig. 11. Schematic of measuring process for output light intensity uniformity.

temperature of the chamber etc. In coating process, the film made from slot-die coater is affected by the fluid properties of the solution, especially the viscosity. The viscosity of the solutions used in the trial runs of MK-20 ranges from 0.25 to 4.3 cp (centipoise) in which the solution behaves as a Newtonian fluid. This indicates that MK-20 can manufacture Perovskite cell with different chemical composition having similar fluid properties of Newtonian fluid. In thermal annealing process, latent heat (evaporation heat) of the solution plays important role in addition to the coated wet film thickness. The latent heat (evaporation heat) of the solution used in the present study spans a large range from 401 to 845 J/kg. Actually, the rest time of the coated wet film also plays important role for obtaining a proper dry film before heat treatment and should be carefully tuned.

4. Key technologies in large-area Perovskite solar cell production

Many manufacturing techniques for large-area Perovskite solar cell (PSC) were proposed by researchers (Kajal et al., 2018), but only a few reported real application data. In the present study, we developed a once-through manufacturing machine (MK-20) for large-area PSC (80 cm × 80 cm max). We used slot-die coater for thin-film coating and developed an innovative scanner-type fast thermal processor (FTP). MK-20 has been run for two years in repeated trial runs. Many experiences in manufacturing large-area Perovskite solar cells were accumulated. We also found some key technologies which is presented here.

4.1. Thickness control of wet film by slot-die coating and chemical composition tuning

Most published data of small Perovskite cell (< 1 cm) with high power conversion efficiency were obtained from samples made by spin coating technique. Spin coating (Zheng et al., 2017) is a method to place a liquid at the center of a solid surface which is rapidly rotated (1000–5000 rpm) and use centrifugal force and the mass transfer phenomena of liquid–vapor interface to spread and produce films of

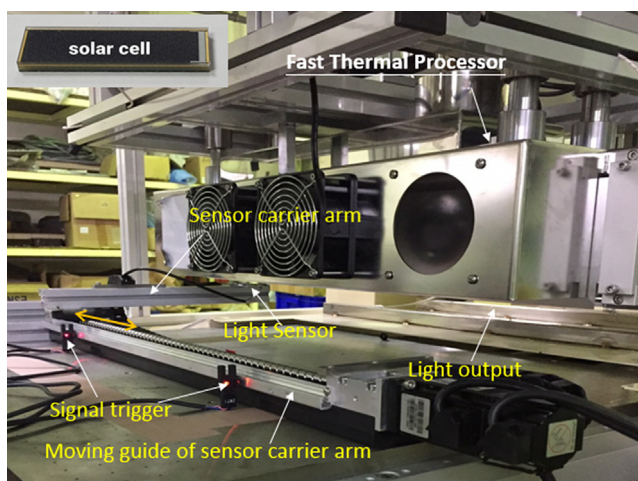


Fig. 12. Device to measure the output light intensity uniformity.

et al., 2020).

This machine can be used to manufacture Perovskite cell using different kind of materials, but needs proper tuning on operating parameters such as substrate moving speed, solution concentration/composition, power of IR heater, and time of heat treatment, air

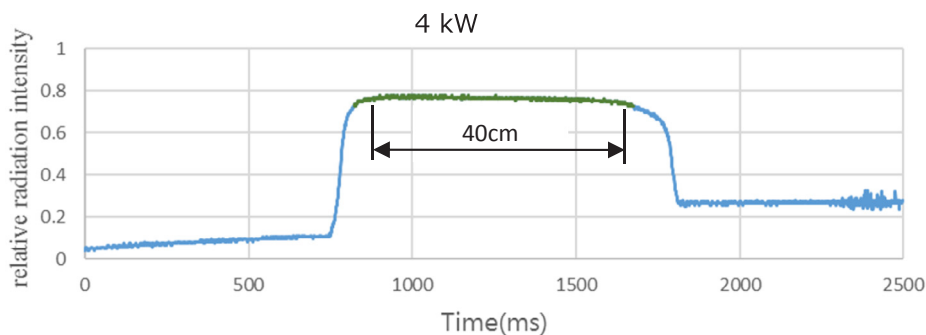


Fig. 13. Variation of output light intensity along the port.

Table 2
Measured uniformity of light output.

	IR lamp 1 kW		IR lamp 2 kW		IR lamp 4 kW	
	Passing time of sensor (s)	Lighting range (cm)	Passing time of sensor (s)	Lighting range (cm)	Passing time of sensor (s)	Lighting range (cm)
Intensity variation < ± 2.5%	0.62	31.0	0.76	38.1	0.85	42.6
Intensity variation < ± 5%	0.70	35.0	0.85	42.4	0.93	46.3
Intensity variation < ± 7.5%	0.73	36.6	0.9	44.8	0.96	48.2
Intensity variation < ± 12.5%	0.78	39.0	0.94	47.2	0.99	49.7
Intensity variation < ± 15%	0.79	39.7	0.96	48.1	1.00	50.1

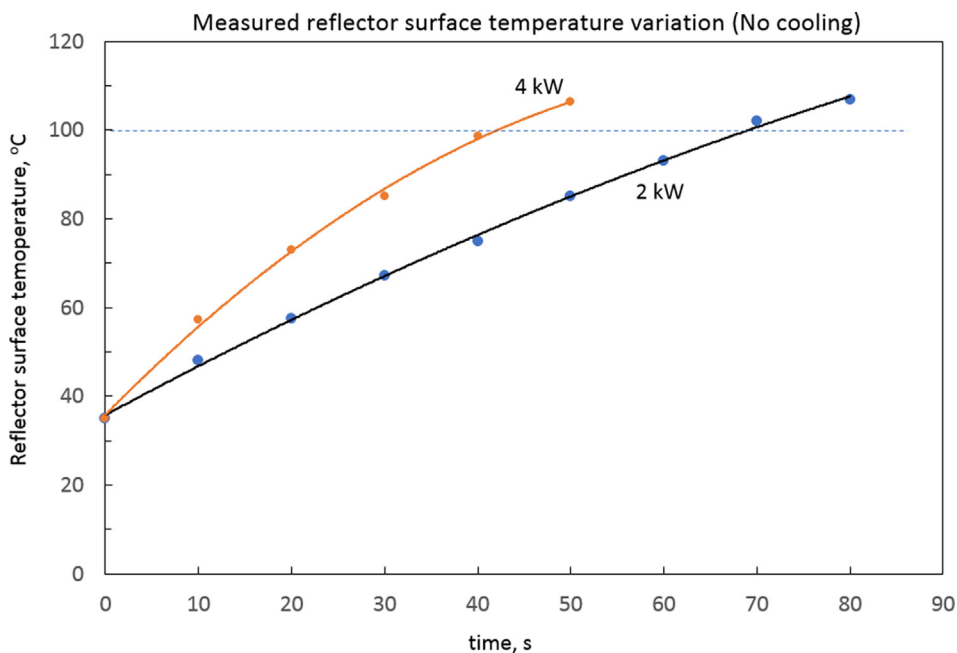


Fig. 14. Variation of reflector surface temperature without fan cooling.

1–5 μm in thickness. The properties of solution will vary along the radial direction under severe convective heat and mass transfer induced by the strong swirling air flow. The variation of chemical and physical properties of solution starts from dilute solution to partly-saturated and fully-saturated solution and finally end up as dried film (with precipitation), as shown in Fig. 22(a). Usually, a dry thin film (1–5 μm) is finally obtained. The heat treatment of this spin-coated dry thin film (like a solid) can be well controlled to obtain a desired final thickness.

Using a precise control in solution feeding flow rate and moving speed of the substrate, we can use the slot-die coater (Fig. 22(b)) to

obtain wet thin film in thickness around 1–5 μm (Fig. 6) very close to spin coating. In this case, when the substrate is moved to the rest area of the machine (Fig. 2) for certain period of time, homogenization and drying process of the coated wet film will occur simultaneously. The wet film is dried from dilute solution, through saturated solution and then becoming dried film (with solid particles precipitation). The final dry film thickness will probably be thinner than that of spin coating. And the power conversion efficiency of the perovskite cell made by spin coating cannot be repeated due to final thickness change.

There are two alternatives in solving this problem: (1) For the case

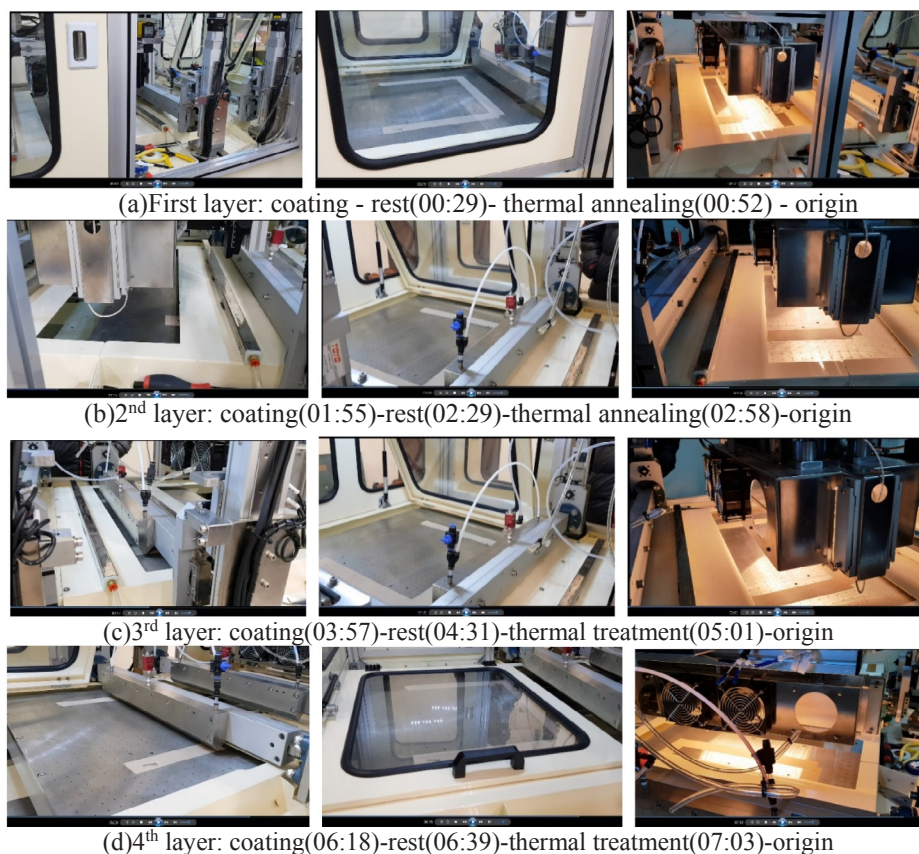


Fig. 15. Automatic operation of the machine.

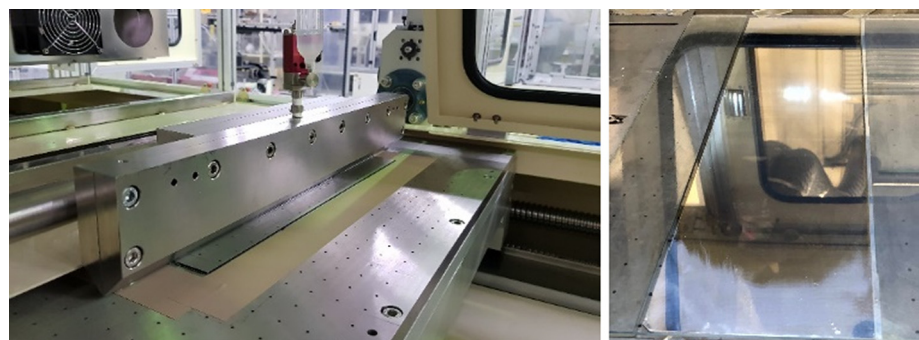


Fig. 16. Coating and heat treatment process (40 cm × 20 cm).

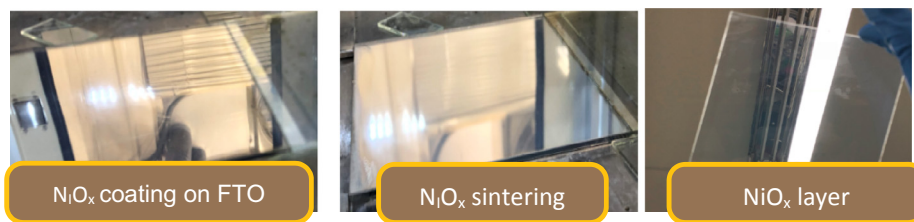


Fig. 17. NiO_x (HTL) layer coating and sintering.

using the same solution concentration as used in spin coating, we have to adjust the coated wet film thickness in slot-die coating such that the final dried film approaches that of the spin coating; (2) changing the solution concentration such that the final dried film approaches that of the spin coating. Some researcher found that using solution concentration about 1/5 of that of spin coating solution will give the same result as spin coating (Deng et al., 2015). In the present study, we found that the ratio of 1/3 is suitable. Trials and errors on these two factors is

necessary in order to obtain the best solar cell efficiency.

4.2. Thickness of coated wet film

It is noted that the film thickness made by slot-die coater should be as small as possible. Thin film will make the molecular force between the liquid and the solid surface larger than the viscous force of the liquid. During heat treatment, thick wet film will create internal flow

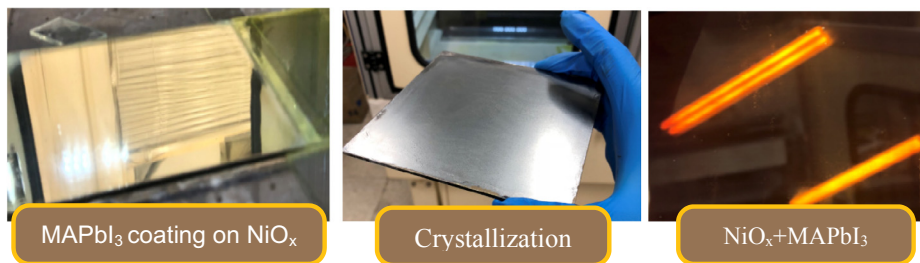


Fig. 18. MAPbI₃ coating and crystallization on NiO_x layer.

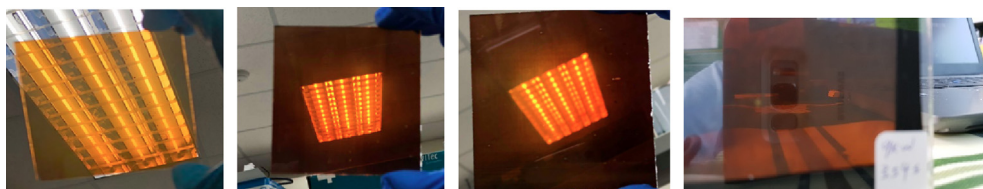


Fig. 19. Finished perovskite cells with 4 layers coating (12 cm × 12 cm).

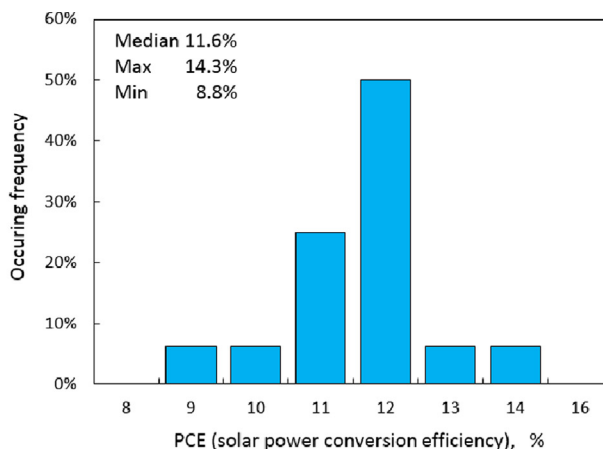
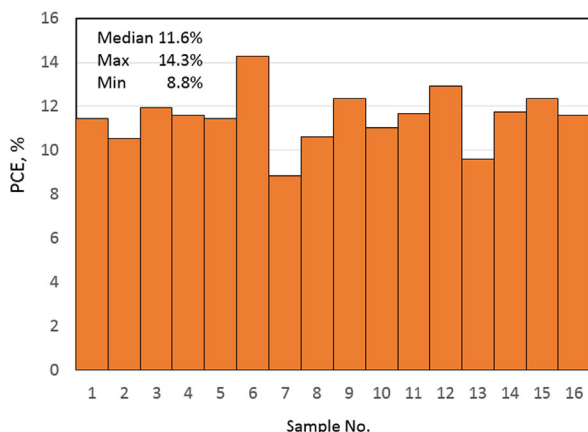


Fig. 20. Solar power conversion efficiency of produced perovskite cells (12 cm × 12 cm).

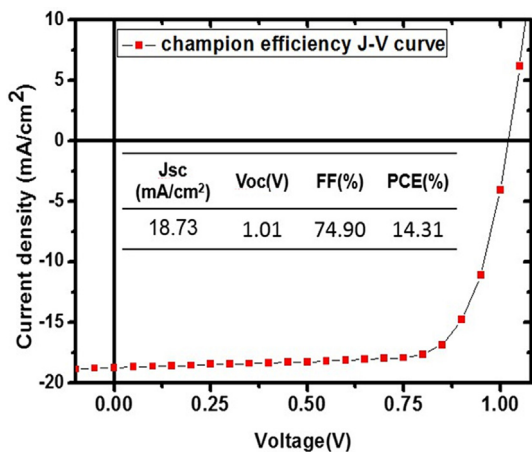


Fig. 21. I-V curve of perovskite cells produced.

phenomenon such as Bénard–Marangoni instability (Sobac et al., 2019). The hexagonal convection cells can develop during the drying process (solvent evaporation) with solid particle precipitation in a volatile solvent and may persist until the film consolidation. This leads to the non-uniform films with surface corrugations (Sobac et al., 2019).

4.3. Pretreatment of wet film before heat treatment

In case the wet film is coated thick, the pretreatment before heat treatment becomes very important. The rest area inside the machine as shown in Fig. 2 (left) provides a space for the coated wet film to rest not only for surface homogenization but also for the wet film to be slowly dried before entering the FTP for fast heat treatment. This will prevent non-uniform film and surface corrugations as described above. An environmental control of the working chamber is required in order to slow down the heat and mass transfer of the wet film. This implies that the small rest area inside the machine has the function of a long furnace as long as the rest time and the environment are controlled adequately. This is why the volume of MK-20 can be so compact. The final dry film thickness obtained is 100 nm for NiO_x layer, 350–400 nm for perovskite layer, 100–150 nm for PCBM layer, and around 15 nm for TBAOH layer. The variation of the total thickness over the 12 cm × 12 cm cell is around 550 ± 50 nm.

4.4. Control technology in thin film coating

Thin film coating can be achieved using slot-die coater and precise control of substrate moving speed and solution feeding flow rate. It was found that the surface tension of the liquid film plays important role in coating process. Surface tension provides a tensile force to draw the film out of the slot die and to attach on the substrate solid surface. The film may break if the force between the surface tension and the liquid

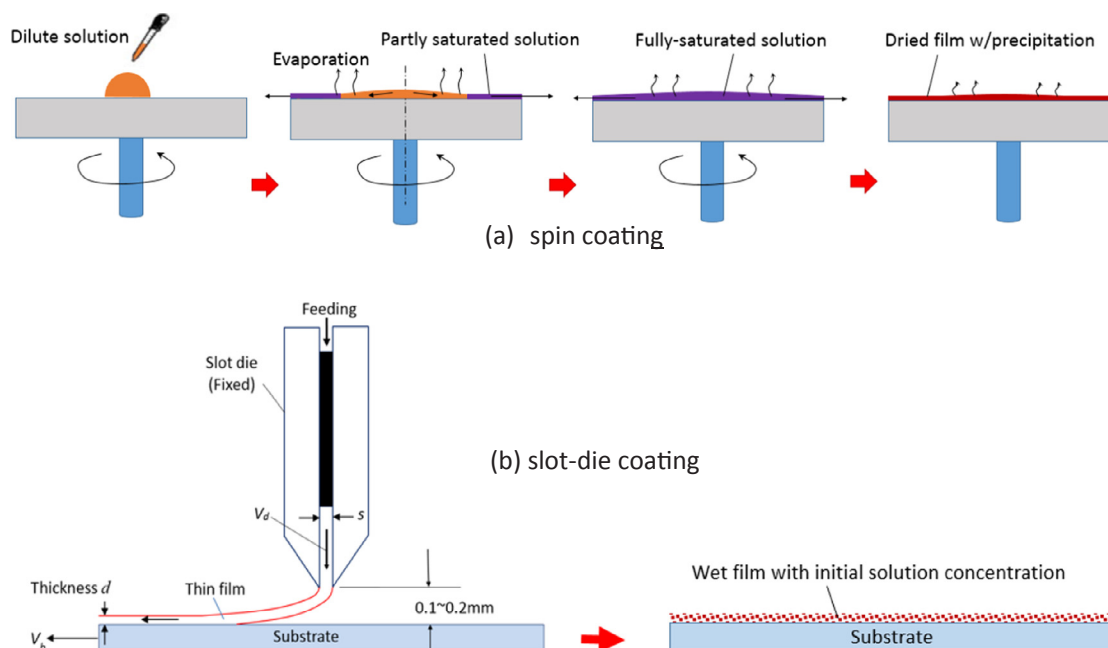


Fig. 22. Sequential process steps in (a) spin coating and (b) slot-die coating.

shear force is not balanced. We also found that the surface tension of some solutions used in the Perovskite cell varies sensitively with temperature. Therefore, a precise control in working chamber temperature, substrate moving speed, and feeding flow rate is very important.

4.5. Spectrum matching of IR heater and heat treatment chemical process

The fast thermal processor (FTP) developed in the present study uses instant IR heating to raise the coated film temperature quickly for reaction. It takes only a few seconds in the heat treatment and the energy consumption is greatly reduced. However, the material properties during heat treatment may be quite different for different radiation heat transfer design. The light absorption coefficient of different layers is not the same. Usually, the IR heater has fixed its output light spectrum in IR region. However, the light absorption coefficient of MAPbI₃ (perovskite layer), for example, is in the region of visible light spectrum. MAPbI₃ almost cannot directly absorb the light from the IR heater. The absorption coefficient of FTO is higher at IR spectrum. Hence, the heat of thermal annealing is indirectly from the heat absorbed by FTO layer. That is, the crystallization of MAPbI₃ is by the indirect conduction heat from FTO.

If the IR heater is replaced by other heat source with light output in visible spectrum, the radiant heat will be directly absorbed by MAPbI₃ and the energy consumption can be further reduced. In this case, the crystallization of MAPbI₃ may involve photo-chemical reaction and the final result may also change. It is noted that the nucleation and crystal growth process during perovskite crystallization is quite critical in large-scale manufacturing of high quality perovskite films (Huang et al., 2019). This requires further research.

4.6. Dynamic heat transfer problem in thermal annealing

It is noticeable that the heat transfer process in the thermal annealing is dynamic (time-variant) since the radiation absorption coefficient of perovskite layer MAPbI₃ may change, from transparent at beginning wet film form to final brown color after crystallization. Therefore, the prediction of surface temperature rise in Fig. 9 will be inaccurate. This complicates the annealing process and worth further studies. The dynamic heat transfer phenomenon also occurs during the heat treatment of other layers. In practice, this can be solved by trials

and errors on the manufacturing process.

5. Conclusion

Perovskite solar cell made from solution process can generate electricity at low cost and is recognized as the next-generation solar cell. However, the manufacture of large-area perovskite solar cells are very few. We developed an innovative Perovskite solar cell manufacturing equipment (MK-20), using once-through process which can continuously carry out thin-film coating and fast heat treatment for four thin layers. The design of slot-die coater and fast thermal processor (FTP) are presented. The manufacturing process is fully automatic and can make small-quantity sample production in good uniformity and high yield rate. The maximum area which can be produced is 80 cm × 80 cm. The maximum production rate is 6 cells per hour. The trial run of MK-20 using for a typical p-i-n type Perovskite cell can achieve 14.3% power conversion efficiency which is about 77% of small cell produced by spin coating and hot plate annealing. The manufacturing process does not use high-precision or expensive equipment. This machine MK-20 is low-cost and able to manufacture various perovskite cells with different chemical composition having similar fluid properties of Newtonian fluid as long as the manufacturing parameters are tuned. Some key technologies in large-area Perovskite cell production were found and also presented.

Declaration of Competing Interest

The authors declared that there is no conflict of interest.

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