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Comparative analysis of mechanical properties of Si substrates processed by different routes

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The purpose of this work is to check the potential of innovative processes for the Si wafers production toward the solar cell industry.^{Q1} Studies have been focused on a comparative analysis of mechanical properties of such wafers, since: (i) reduced wafer strength leads to a high breakage rate during subsequent handling and solar cell processing steps, (ii) cracking of solar cells has become one of the major sources of solar module failure and rejection. Therefore while developing new types of wafer materials and processing, it is essential to assess the mechanical strength of the wafers. Mechanical properties of several innovative Si based substrates are estimated. The bending strength measurements of the silicon wafer are performed using the ring-on-ring set-up coupled with a numerical model to obtain estimate of the fracture stress and the Weibull parameters of the fracture distribution. Results are presented for five different materials: sintered Si powder, standard multi-crystalline Si, Czochralski monocrystalline Si, and two types of thermal sprayed Si wafers.

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1 Introduction Currently, the PV market is dominated by crystalline silicon solar cells, and about 40% of the silicon 1 module cost is from the silicon wafers. Therefore, a major part 2 of current research activity is concentrated on a search for 3 4 alternative silicon based solar cell concepts with reduced consumption of high-purity silicon. In conventional Si wafer 5 6 based solar cell technology, most of the Si material acts as a 7 mechanical carrier for the solar cell structures. However, since most of the optical absorption in Si takes place in the upper 15-8 9 $30 \,\mu$ m, it is sufficient to use only thin Si layers with thicknesses 10 in this range. Indeed, if special optical confinement schemes are implemented, even thinner layers can be used [1]. In general, 11 thin-film photovoltaics are assumed to become a market 12 dominating technology in the long term and any development 13 in this field is extremely important for the PV industry [2]. 14

To be able to reduce thin Si film solar cell cost, both 15 material and material fabrication costs must be reduced. This 16 can be achieved by growing a high quality "expensive" thin 17 active crystalline Si layer onto a less expensive substrate. 18

Ceramic or glass based materials have been proposed as such 1 substrates. Such option is still envisaged to be cheaper than 2 the use of conventional Si-wafer substrates, as thin film 3 PECVD and PVD deposition processes, for example, have 4 been routinely used to produce high quality thin films for 5 electric and optoelectronic devices at accepted consumer 6 costs. Depending on process conditions, thin Si based-layers 7 of amorphous (a-Si), hydrogenated amorphous (a-Si:H), 8 microcrystalline (µc-Si), or polycrystalline (poly-Si) silicon 9 can be grown on such substrates. For µc-Si-based solar cell 10 structures deposited on glass substrates, energy conversion 11 efficiencies up to 10% have been demonstrated [3]. At 12 the same time solar cells utilizing thin-film polycrystalline 13 Si with an optimum thickness about $20 \,\mu\text{m}$ can achieve 14 photovoltaic power conversion efficiencies greater than 19% 15 [4]. However, the use of ceramic and glass substrates for 16 thin Si solar cells have some problems: 17

Conductivity: Glass and ceramic substrates are non- 18 conductive. Thus, a lot of attention has been directed towards 19



making a highly conductive back side electrode as well as a 1 system for contacting the electrode after deposition of silicon 2 [5]. Diffusion of aliovalent dopants from the electrode is a 3 4 big problem. Conducting SiC is being developed as an 5 alternative [6]. However, still a number of problems have to 6 be solved in this approach, since crystallization of Si layers 7 on SiC substrates is rather problematic.

8 Lattice matching: Glass and ceramic substrates have no 9 crystallographic relation to silicon to aid crystallization at lower temperatures and/or into higher crystallographic 10 quality. Thus, higher temperatures are needed when using, 11 e.g., a higher cost single crystal Si wafer. 12

Purity: Care must be taken to avoid diffusion of dopants 13 into Si during deposition and post-processing. 14

Temperature stability: One needs >700-1000 °C for 15 growth of poly-Si. This is marginal for the case of glass 16 substrates. Therefore lower temperatures are used for such 17 processes. 18

It can be concluded that there is a demand for advanced 19 low-cost substrates, which can be used for thin Si-based 20 solar cell structures. So far such low-cost Si supporting 21 22 substrates have been based on highly doped Si wafers 23 processed in the same way as conventional Si wafers, i.e., by crystallization of ingots and wafering. Such 24 25 conventional processing of Si wafers can be substituted by a cost-effective powder-to-wafer processing using 26 ceramics technology, hence avoiding costly crystallization 27 and wafering steps. Such "powder-to-wafer" approach, 28 can simplify the wafer based processing of the supporting 29 Si substrates and therefore relevant thin Si-based solar 30 cells, Usage of a low grade Si feedstock can reduce the 31 cost of Si wafers even further. These low purity substrates 32 can be processed from a low-quality Si powder. When 33 the powder is shaped and sintered into an appropriate 34 substrate, it can be considered as a poly-Si seeding material, 35 36 which can provide good crystallization conditions for any Si-based layers deposited and annealed at appropriate 37 conditions. 38

39 Such substrates have several advantages compared to ceramic or glass substrates: (i) highly doped silicon is 40 conductive. Hence, the substrate can be used as electrode, 41 42 avoiding any contact problems; (ii) a perfect lattice match will lead to the lowest possible crystallization temperatures 43 for the deposited high purity thin film, (iii) silicon-based 44 45 low-cost supporting substrates are fully compatible with the 46 deposition/crystallization processes of thin Si layers on top of such substrates. 47

The concept of crystalline silicon thin-film solar 48 cells on low-purity substrates provides a hope to reduce 49 substantially the consumption of high-purity silicon 50 material and has at the same time potential to reach high 51 52 efficiencies comparable to wafer silicon solar cells. The goal of this article is to test mechanical properties of Si 53 wafers, processed in frame of an innovative approach 54 based on a thermal spray of Si powder and sintered Si and 55 to compare both Si powder-based substrates with those, 56 which are processed by casting or CZ growth methods. 57

This article firstly describes the material and samples preparation. Secondly the electrical properties are quickly 2 studied. The measurements and the associated numerical 3 model are presented in the next section. Finally the results 4 are discussed as well as possible explanations for the 5 discrepancies in the material properties and their link to 6 processing routes. 7

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2 Samples material and preparation

2.1 Materials and processing techniques The 9 original powder (Si feedstock) for the substrates comes from 10 a by-product stream of the Elkem Solar metallurgical process 11 route to produce Elkem Solar Silicon[®] in a form of Si 12 powder. The quality is in-between the process starting-point: 13 metallurgical grade silicon and the end-point: solar grade 14 silicon. Several types of Si powders in the wide range of 15 particle size distribution (PSD) have been tested. The PSD 16 values were varied in the range of $1-150\,\mu\text{m}$ and were 17 adjusted to the special requirements of each processing 18 techniques used. The following techniques, which utilized 19 as produced Si powder directly without any additional 20 processing or cleaning (like HF dip) steps have been tested: 21 (i) casting of Si ingot from Si powder using a pilot scale 22 vertical gradient freeze (VGF) furnace and (ii) thermal spray 23 of Si powder. Important to underline that in both cases 24 Si powder was covered by native oxide and processing 25 conditions were optimized to reduce this oxide upon the 26 fabrication. In case of a thermal spray technique PSD values 27 were below 100 µm. As-processed Si powder was used 28 for sintering of Spray-1 Si substrates. For the other type 29 of sprayed Si substrates 3% (weight) of Al powder was 30 mixed with the Si powder-based feedstock prior the spraying 31 (Spray 2 substrates). 32

In case of the casting process, which was carried out 33 using a VGF furnace at SINTEF, Si powders with PSD 34 values up to 150 µm have been used. The silicon powder-35 based feedstock was melted and solidified. The thermal 36 conditions and mass transfer in this furnace have been 37 studied thoroughly the latest years [7, 8]. This required 38 special care during furnace operation. 39

Since the resistivity of the final substrates were supposed to be relatively low ($<0.01 \Omega$ cm), 1.5 g of 95– 97% pure boron was added to the charge before melting. Even though – according to phase Si-boron diagrams [9] the melting point of pure boron is about 2100 °C, the boron will dissolve completely in the liquid silicon at 1430 °C since the solubility is about 10% and the added amount is merely 160 ppm.

The logged data from the casting are shown in Fig. 1.

A complete description of the operation of the furnace under normal conditions has been given earlier [10]. The main differences between standard furnace operation and the one done in this study are:

1. Double layer of coating was used to ensure no sticking between ingot and crucible even with long time at high temperature.

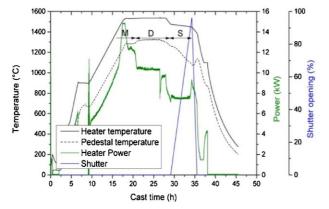


Figure 1 (online color at: www.pss-a.com) Data from the Crystalox furnace during casting. Shown are the heater temperature, pedestal temperature (temperature beneath crucible), heater power, and shutter opening during crystallization. M, D, and S denote melting, dissolution of boron and mixing, and solidification phases, respectively.

- 2. Since the feedstock was in the form of powder, the initial pump-down phase was done very gentle in order to avoid sucking powder out of the crucible.
- 3. Gas flow after the vacuum phase was reduced in order not to blow feedstock out of the crucible.
- 4. After complete melting, the melt was held for 6.5 h longer than normal in order to secure complete dissolution of the boron.

2.2 Wafer samples preparation Highly conductive p+ Si wafers from casted Si ingots were obtained by wire sawing.

Additionally series of Si wafers were produced by thermal spray route, with thicknesses between 300 and 1000 μ m, and dimensions 65 × 50 mm² have been prepared from nominally un-doped low-cost Si powder. Silicon layers (wafers) were detached from specially selected and prepared substrates/ moulds without breaking (Fig. 2). For the mechanical properties measurements sintered wafers were cut on $1 \times 1 \text{ cm}^2$ smaller pieces by laser. Summary of samples material and process routes can be seen from Table 1.



Figure 2 (online color at: www.pss-a.com) Si wafers ($65 \times 50 \text{ mm}^2$) fabricated by thermal spray.

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 Table 1
 Summary of samples material and process routes.

sample name	material	process route
Si powder-based multi	Elkem powder	ingot sintering/VGF furnace
P-type multi	solar grade Si	conventional casting
as cut P-type C7	solar grade Si	Czochralski growth
Spray 1	Elkem powder/100% Si	thermal spray
Spray 2	Elkem powder	thermal spray
	3% Al/97% Si	

3 Measurement of electrical properties Resis-1 tivity measurements have been performed by the four-probe 2 method. It has been found that Si wafers fabricated from 3 the casted Si ingot have resistivity $<0.01 \Omega$ cm. Such 4 wafers can be used as highly supporting ones for thin 5 Si-based solar cells. Si wafers processed by thermal spray 6 from nominally non-doped low-cost Si powder demon-7 strated resistivity in the range of $1-10\,\Omega$ cm, which shows 8 that sintering process upon thermal spray has been done 9 properly and no barriers between grains have been created. 10 Since this work is focused on mechanical properties of Si 11 powder-based substrates, doping issues for thermal sprayed 12 substrates were not addressed. However, it can be noted 13 that processing of such wafers using highly doped Si 14 powder should result in highly doped and therefore highly 15 conductive substrates. 16

4 Measurement of mechanical properties

4.1 Experimental set-up In order to assess the 18 mechanical strength of the wafer produced by the new 19 process routes, the ring-on-ring test was used. Based on the 20 recommendations of the ASTM standard C1499-08 [11], 21 a smaller experimental set-up has been designed as 22 illustrated in Fig. 3. This set-up has been selected to measure 23 the material intrinsic strength and avoid the effect of edge 24 defects. 25

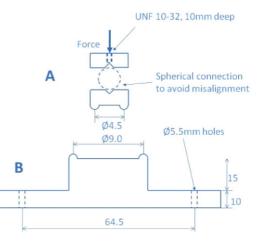


Figure 3 (online color at: www.pss-a.com) Schematic view of the experimental set-up.

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4.1.1 Testing equipment The first tests were per-1 formed in the Bose Electroforce 250N test machine on 2 lower speeds (i.e., $0.1-0.2 \text{ mm min}^{-1}$). Due to irregular 3 loading rate at the lower speeds, the speed was increased to 4 1 mm min^{-1} . Nevertheless irregular jumps of the machine 5 remains and it led to machine change for the rest of the study. 6 7 Therefore only Si powder-based multi samples have been 8 tested using Bose Electroforce 250N machine. An Instron 9 2kN test machine was used instead in all the remaining experimental work presented in this paper (P-type multi, 10 As cut P-type C7, Spray 1 and Spray 2). 11

The fixture was made of heat-treated steel, hardened to 12 about 40 HRC. 13

The data acquisition sampling rate was 100 Hz for the tests 14 done in the Bose machine and 15 Hz for the Instron machine. 15

16 **4.1.2 Calibration** Both test machines were recently calibrated by the manufacturer. Due to limited utilization of 17 the load cell of the Instron machine an additional control was 18 performed with one of the reference load cells of the test 19 laboratory. Check of coaxiality of the test fixture was done 20 during mounting, both by visual inspection and by using pre-21 machined pin holes in the center of the upper and lower 22 fixture and a guiding pin. The plane parallelism of the upper 23 24 and lower ring was corrected before each test by visual 25 inspection of a light gap of about 0.25 mm between the 26 upper and lower ring. The light gap was checked in two 27 perpendicular directions. The fixture was thoroughly cleaned 28 before each test in order to prevent silicon debris deposition on the ring surfaces. 29

30 4.2 Numerical model A numerical model of the ringon-ring mechanical test has been established. The main 31 objectives were to assess the uniformity of the stress 32 33 distribution inside the loading ring area (especially at the contact point) and develop a formula to compute directly 34 35 the maximal principal stress for the displacement and the 36 sample thickness.

37 The model was developed using Abaqus/Standard 6.11 38 with implicit time integration. Only a quarter of the sample and rings is modeled (see Fig. 4). The sample mesh size is 39



Figure 4 (online color at: www.pss-a.com) Abaqus model of the ring-on-ring test.

0.05 mm. The material properties are indicated in Table 2. The dimensions and element type are provided in Table 3.

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As illustrated in Fig. 5, considering the thinnest sample which is the most critical in terms of stress distribution, the stress inside the loading ring is quite homogeneous and a relatively small increase (\sim 5%) is observed only in the contact area with the loading ring. Therefore the dimensions of the sample and the rings are adequate for wafer strength measurements.

By applying the model to various thicknesses (0.20, 0.39,0.6, and 0.8 mm) and performing a regression analysis, we obtained the following formula:

$$\sigma = (2616h + 3067)\delta^2 + (14\,830h - 465)\delta,\tag{1}$$

Table 2 Silicon properties for the numerical model.

material	silicon
model Young's modulus Poisson's ratio	isotropic elasticity 162.5 GPa 0.223

 Table 3 Numerical model dimensions and element type.

part	shape	finite element model
silicon specimen	square ^a (10×10)	8-nodes brick element
loading ring	ring ^b (4.5×0.5)	shell, discrete rigid
support ring	ring ^b (9×0.5)	shell, discrete rigid

^aLength and width in mm.

^bDiameter and curvature radius in mm.

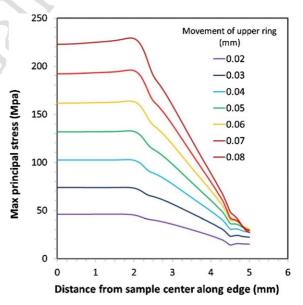


Figure 5 (online color at: www.pss-a.com) Stress distribution along the cross-section of the sample (thickness: 0.2 mm).

2 where σ is the stress at the sample center in MPa, *h* is sample 3 thickness in mm, and δ is the displacement in mm.

4 **5** Results and analysis The raw data from the series 5 of mechanical tests using the ring-on-ring set-up previously described are presented in Figs. 6-10. The displacement of 6 7 the ring is given in the x-axis while the y-axis provides the 8 applied force measured by the load cell. As mentioned in 9 the Section 4.1, the curves of the initial set-up (Fig. 6) using 10 the Bose Electroforce 250N test machine has a stair case appearance. Nevertheless it does affect neither the curve 11 shape nor the displacement at fracture. The main issue in this 12 series of test is the large variation in the slope of the curves 13 14 before the peak force value is reached. We would expect for 15 such material (except maybe for the spray samples) a very reproducible elastic response. Indeed we have previously 16 17 measured the strength of the Si powder based multi samples

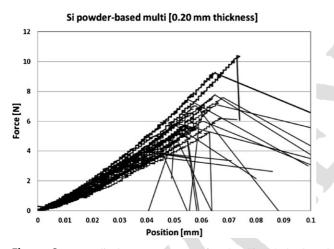


Figure 6 Force displacement curves for the Si powder-based multi samples (28 samples).

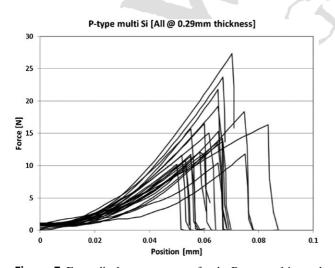


Figure 7 Force displacement curves for the P-type multi samples (23 samples).

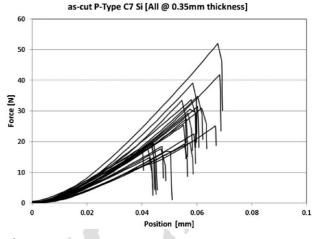


Figure 8 Force displacement curves for the As cut P-type C7 samples (25 samples).

using the 4pt-bending test and the curves were overlapping. 1 The reason for this discrepancy is not clearly identified but 2 there are two possible explanations. First, as mentioned by 3 Cotterell et al. [12], the proper positioning of the samples for 4 the ring-on-ring test could be an issue. Friction between the 5 ring and the sample can lead to inaccurate results. Therefore, 6 Wasmer et al. [13] recommend the use of carbon paper. 7 Please note that the Weibull fit of the experimental data 8 without carbon paper from Wasmer et al. shows a similar 9 staircase aspect as some of our results (especially Fig. 12 10 where one can observe a slope change for the experimental 11 curves between a probability of 0.4 and 0.5). It is also 12 important to note that this friction effect might be dependent 13 on sample roughness which varies between our materials. 14 Nevertheless we assume that these effects induce only a 15 systematic error on the force measurements. Therefore the 16 analysis of the results based on the displacement should 17

Spray layer 1 [All @ 0.56-0.65mm thickness]

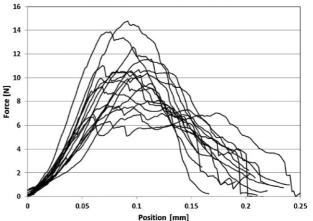


Figure 9 Force displacement curves for the Spray 1 samples (16 samples).



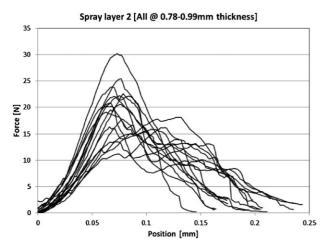


Figure 10 Force displacement curves for the Spray 2 samples (16 samples).

provide good estimate of the samples strength for comparison between different materials.

The raw data has been post-processed using Eq. (1). Then the stress values at fracture were ordered and related to a probability in order to build the Weibull distribution.

Indeed the standard parameters in the literature to assess the wafer strength are the parameters based on the Weibull distribution.

The probability of fracture is described by an exponential function with two parameters:

$$P(\sigma) = 1 - e^{-(\sigma/\sigma_0)^m}.$$
(2)

From the ordered values, a curve fitting procedure has been applied in Excel. The results for the three first materials are presented in Figs. 11–13. In addition, previous results with Si powder based multi samples obtained with 4ptbending set-up and larger samples $(5 \times 5 \text{ mm}^2)$ are also presented for comparison.

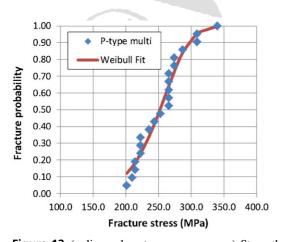


Figure 12 (online color at: www.pss-a.com) Strength measurements and Weibull fit for P-type multi samples ($\sigma_0 = 264$ MPa and m = 7.5).

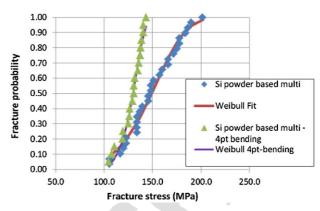


Figure 11 (online color at: www.pss-a.com) Strength measurements and Weibull fit for Si powder based multi samples using ringon-ring test ($\sigma_0 = 158$ MPa and m = 5.9) compared with 4pt-bending experimental measurements ($\sigma_0 = 133$ MPa and m = 14).

As illustrated by Figs. 9 and 10, the material behavior of 1 the spray samples is significantly different. Therefore the 2 direct application of the previous procedure is not possible 3 and the study of the sprayed samples requires a more detailed 4 analysis of the material. In all the previous cases we assume 5 that the material properties are similar to bulk silicon 6 which is a quite good approximation. Nevertheless for 7 the sprayed samples, the microstructure of the material is 8 non-homogeneous which is mainly due to the presence of 9 porosity and layers. 10

In order to obtain a better estimate of the equivalent fracture stress for this material, we will need to first estimate the porosity and subsequently estimate the relative correction of the stress value resulting from this porosity. As illustrated on Fig. 14, the metallography image for Si sample Spray 1 is processed using a simple threshold method in order to detect the holes and estimate the porosity.

This procedure is not the most advanced and provides only18an estimate of the porosity. Using this technique we obtain a19

1.00 As cut P-type C7 0.90 0.80 Weibull Fit Fracture probability 0.70 0.60 0.50 0.40 0.30 0.20 0.10 0.00 100.0 150.0 200.0 250.0 300.0 350.0 400.0 Fracture stress (MPa)

Figure 13 (online color at: www.pss-a.com) Strength measurements and Weibull fit for P-type multi samples ($\sigma_0 = 283$ MPa and m = 6.4).

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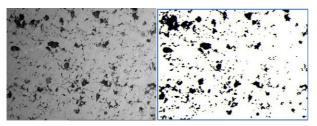


Figure 14 Comparison between initial metallography image $(\times 500)$ on the left and processed image for porosity estimate on the right.

value of 11% porosity, which agrees with the results obtained for other type of powder based materials (yttria-stabilized zirconia coatings) processed by thermal spray [14].

4 In the work by Wang et al. [14], different spraying 5 techniques are used with increasing level of porosity and the effect on the elasticity modulus for each sample is measured. 6 7 Two values are closed to our porosity estimate: 7.6% and 12.2% porosity for which the elasticity modulus is reduced to 8 45% and 25% of the bulk value, respectively. From these 9 10 values, we obtain by simple interpolation a decrease to 30% 11 of the elasticity modulus of bulk silicon. Various materials sprayed using high velocity oxygen fuel technique (HVOF) 12 13 ranged also between 30% and 40% of the bulk elastic 14 modulus [15].

Therefore, in order to estimate the fracture stress using 15 the previous formula, we apply a corrective multiplicative 16 17 factor of 0.3 to the fracture stress computed from the displacement at rupture (Eq. 1). The results are presented in 18 19 Figs. 15 and 16. Please note that these values are sensitive to the sample thickness (not precisely measured over the whole 20 surface of the sample and to the porosity (estimated for only 21 one sample) and therefore this analysis will provide only 22 a coarse estimate of the material strength. In any case, as 23 24 presented by Margadant et al. [16], it is very difficult to 25 obtain an accurate value of the elastic modulus of a coating as it depends on the measurement and spraying technique used. 26

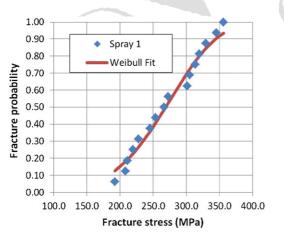


Figure 15 (online color at: www.pss-a.com) Strength measurements and Weibull fit for P-type multi samples ($\sigma_0 = 290$ MPa and m = 4.8).

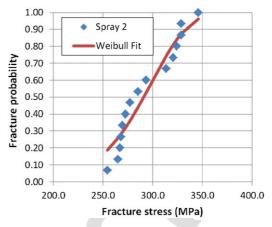


Figure 16 (online color at: www.pss-a.com) Strength measurements and Weibull fit for P-type multi samples ($\sigma_0 = 303$ MPa and m = 9).

Table 4 Summary of the Weibull fit of the ring-on-ring tests(or 4pt-bending when specified).

material	Weibull mean stress, σ_0	Weibull variance, <i>m</i> 5.9		
Si powder based multi	158			
Si powder based multi	133	14		
(4pt bending)				
P-type multi	264	7.5		
as cut P-type C7	283	6.4		
Spray 1	290	4.8		
Spray 2	303	9		

For comparison the results for the five materials are 1 summarized in Table 4. As expected the variance for all 2 materials is in the same range except for the 4pt-bending test 3 where usually a larger variance is obtained. 4

It is also worth mentioning the significant increase 5 between Spray 1 and Spray 2 samples. This might be 6 explained by an influence of 3% of Al powder, which is 7 mixed with Si powder based feedstock on mechanical 8 properties of sprayed Si wafers. More detailed studies are 9 required to investigate this effect. 10

6 Conclusions Comparative analysis of mechanical 11 properties of Si samples obtained from new process routes 12 for the production of silicon wafers has been presented. 13 Characterization of the mechanical strength of the different 14 silicon samples was investigated using a ring-on-ring test. 15 The maximum principal stresses at failure during bending 16 were calculated to indicate the fracture strength and fitted to a 17 Weibull distribution. The study showed that: 19

 In spite of some experimental challenges, the ring-on-ring set-up provided acceptable estimate of the material for various materials and process routes.

- The Si powder based multi samples were the weaker but
 still with a reasonable strength comparable to standard
 wafer without etching.
- The spray samples display a specific material behavior due to porosity and layering. Nevertheless the final material strength is high.

As a summary, this work has demonstrated the potential of
 new process routes leading to the production of Si wafers with
 adequate material properties both electrical and mechanical.

It can be concluded that Si powder-to-substrate approach can be utilized for the processing of Si based supporting substrates, which potentially are fully compatible with the deposition/crystallization processes of thin Si layers on top of such substrates and posses comparable with multi-Si/Cz-Si substrates mechanical properties.

Further work should be performed to analyze in more details the microstructure of the new materials and assess their downstream integration in the solar cell production.

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- Q1: Author: Please expand the forename of the authors R. Gløckner, M. Syvertsen and also check the telephone number of the corresponding author.
- <u>Q2</u>: Author: As per the style of the journal, et al. is not allowed in the reference list. Please check all the et al. references.
- <u>Q3</u>: Author: Please provide the author names.
- <u>Q4</u>: Author: Please check the presentation of this reference.
- <u>O5</u>: Author: Please provide the page range.

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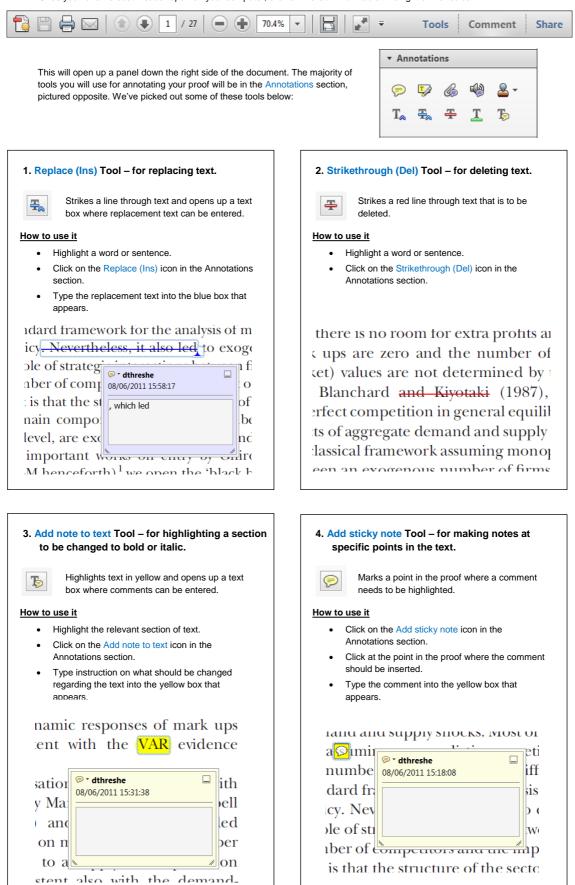
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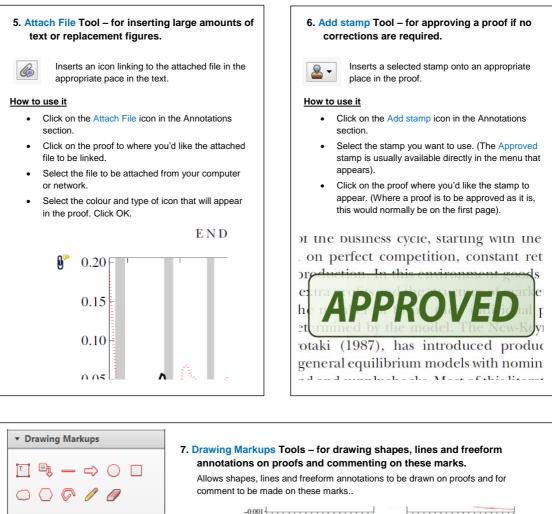
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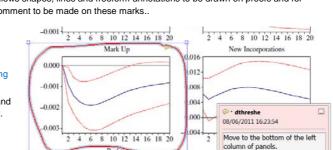
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