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A planar thermoelectric power generator for integration in wearable microsystems

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ABSTRACT

A technique for IC-compatible fabrication of a planar (in-plane) thermoelectric (TE) power generator using a thermopile composed of n-type bismuth telluride (Bi₂Te₃) and p-type antimony telluride (Sb₂Te₃) thin-films is presented. The research demonstrates that the thermal co-evaporation of bismuth/antimony (Bi/Sb) and telluride (Te) is the most suitable deposition technique. The measurements showed TE performance properties of the deposited thin-films that are comparable to those reported for the same materials in the bulk form. The measurements showed absolute values of the Seebeck coefficient in the range 91–248 μ V K $^{-1}$, an electrical resistivity in the 7.6–39.1 μ 0 m range and a thermal conduction between 1.3 and 1.8 W m $^{-1}$ K $^{-1}$. The best resulting figures-of-merit, ZT, at room temperatures were 0.97 and 0.56 (equivalent to power-factors, PF, of 4.87 \times 10 $^{-3}$ and 2.8 \times 10 $^{-3}$ W K $^{-1}$ m $^{-2}$) for the Bi₂Te₃ and Sb₂Te₃ thin-films, respectively. The IC-compatibility and the dependence of the TE performance on technological details, such as photolithography and wet etching used for patterning the thin-films have also been investigated. The converter dimensions for best performance were analysed and a prototype of a planar TE power generator was fabricated.

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1. Introduction

Thermocouples are thermoelectric (TE) devices. Conventional thermocouples based on metal wires are cheap, reliable and widely used for measuring high temperatures. This is the case of furnaces, which are widely used in the microelectronic industry. A thermocouple is a simple electric circuit, formed by two dissimilar conductors joined at both ends (i.e. the junctions). Opening the circuit by cutting one of the wires enables the measurement of a voltage, which is proportional to the difference in temperature at the two junctions (the Seebeck effect). Consequently, the thermocouple can be used to generate a voltage proportional to temperature difference without the need of any external electrical bias. A temperature sensor results if one of the junctions is maintained at a well-known temperature. The thermocouple can also be used as an actuator. Applying an electrical current through the thermocouple allows the transportation of heat from one junction (the cold junction) to the other (the warm junction at ambient temperature). As a result, the cold junction is cooled (the Peltier effect) [1]. The Seebeck effect can be used for both temperature (difference) sensing and for realising heat engines to convert heat into electrical energy. The advantages of TE energy conversion is that moving mechanical parts are avoided, which enables high system reliability, quiet operation and it is usually environmentally friendly. The compact and distributed power is a very attractive feature in a wide range of applications.

However, the TE effect is very inefficient in most materials and the research into special materials is mandatory for practical use. The best performance is obtained in the presence of heavily doped semiconductors, such as the bismuth telluride or the silicon germanium. When using semiconductors, the most desirable situation is when the base materials are both n- and p-doped, since this allows the use of essentially the same material system for fabrication of the two TE legs between the junctions [2]. The selection of materials and the suitable fabrication technologies are further constraint by IC-compatibility requirements in the case of TE power generation in a microsystem, which is the objective of the research presented in this paper. Additional requirements imposed by the microsystem application are: small size, low weight and thermal isolation from the substrate [3].

The functional integration of efficient solid-state TE devices and microelectronic circuits offers many benefits. One is the implementation of local cooling for thermal stabilisation of an on-chip reference element or for reducing leakage current in a critical component such as a photodetector. Another implementation is in TE power generation to enable operation of a low-power circuit without external electric power source, such as a battery. Despite the huge potential of TE self-lowering in autonomous microsystems,

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only few approaches to fabricate such microdevices have been reported up to now [4-7].

Due to its compatibility with IC technology, polycrystalline SiGe alloys and polycrystalline Si are commonly used in thermopile applications. Their use in microcoolers has been investigated; however, their performance is very low when compared to that of tellurium compounds, which have been used for many years in conventional large-area Peltier devices [3]. Tellurium compounds (n-type bismuth telluride, Bi₂Te₃ and p-type antimony telluride, Sb₂Te₃) are well-established room temperature TE materials and are widely employed by the industry in conventional TE generators and coolers. Several deposition techniques have been investigated for their suitability for fabricating thin-films materials. The direct evaporation of the bulk materials for the deposition of Bi₂Te₃ films was demonstrated by da Silva to be non-suitable. The large differences in vapour pressure of bismuth and tellurium resulted in a compositional gradient along the thin-film thickness [8]. Other techniques explored for the deposition of Bi₂Te₃ thin-films are the thermal co-evaporation [9], the electrochemical deposition [10], the co-sputtering [4], the flash evaporation [11] and the metalorganic chemical vapour deposition (MOCVD) [12]. Although all these approaches are in principle suitable, the co-evaporation was used in this work to obtain both the n-type Bi₂Te₃ and the ptype Sb₂Te₃ thin-films, because it allows to precisely control the estequiometry of the deposited thin-film with the lowest costs. Only the MOCVD is better than the co-evaporation to obtain thinfilms with good uniformity and with the desired estequiometry. However, in order to do the deposition of thin-films by MOCVD, a reactor chamber is needed. Thus, this process is too expensive and it requires additional security procedures, when compared with the co-evaporation [13].

In this work, both the n-type Bi₂Te₃ and the p-type Sb₂Te₃ thinfilms were obtained by co-evaporation, yielding devices with a TE figure-of-merit, ZT, of 0.97 and 0.56, respectively. The Bi₂Te₃ selected samples showed a Seebeck coefficient in the range $152-248 \,\mu\text{V}\,\text{K}^{-1}$, resistivity of $10.6-16.6 \,\mu\Omega\,\text{m}$, a thermal conductivity of about 1.3 W m⁻¹ K⁻¹ [14], a carrier concentration \approx 6 \times 10¹⁹ cm⁻³ and a Hall mobility from 80 to 120 cm⁻² V⁻¹ s⁻¹. The EDX analysis (Energy-Dispersive X-ray spectroscopy) revealed the stoichiometric composition of the selected samples. For the p-type thin-films, the best available results (or selected samples) include Seebeck coefficients in the range $91-188 \mu V K^{-1}$, a resistivity of 7.6–39.1 $\mu\Omega$ m, a thermal conductivity about 1.7 W $m^{-1}\,K^{-1}$ [14], a carrier concentration $\approx 4 \times 10^{19} \, \text{cm}^{-3}$ and a Hall mobility from 120 to $170 \, \text{cm}^{-2} \, \text{V}^{-1} \, \text{s}^{-1}$. These values are similar to the best values found in the literature for bulk materials [15]. Since bulk materials are used in conventional macro-scale modules, a similar performance of the microconverter is feasible. The performance of a TE device depends on the figure-of-merit, ZT, of the material, which is given by [3,4,15]:

$$ZT = \frac{\alpha^2}{\rho\lambda}T\tag{1}$$

where α is the Seebeck coefficient, ρ the electrical resistivity, λ the thermal conductivity and T the temperature. Furthermore, the power-factor, PF (W K⁻¹ m⁻²) gives the electric power per unit cross-sectional area of heat flow at a given temperature gradient between the hot and the cold junction. The PF is given by:

$$PF = \frac{\alpha^2}{\rho} \tag{2}$$

The influence of deposition parameters on the thin-film performance has been studied in detail. For obtaining data that is statistically significant, more than one hundred samples were fabricated. One essential parameter in the fabrication of TE microdevices is the film-to-substrate adhesion. To investigate this issue, three

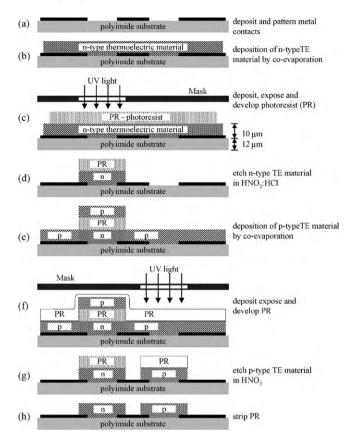


Fig. 1. The fabrication steps of TE devices.

different substrate materials have been used: glass, silicon and polyimide (kapton). However, for actual TE applications, a kapton film was selected as substrate, because of the low thermal conductivity (0.12 W m $^{-1}$ K $^{-1}$). Moreover, the thermal expansion coefficient (12×10^{-6} K $^{-1}$) closely matches the thermal expansion coefficient of the telluride thin-films, thus reducing the residual stress and increasing the adhesion. The information obtained on silicon and glass is important for MEMS-based TE devices, where the micromachining is applied for thermal definition of the microstructure. Flexible substrates enable the integration with many novel types of devices, however, also introduce complications, such as the uncommon mechanical properties of the composite film-substrate.

2. Fabrication

Two different approaches can be used for the on-chip integration of TE devices: the transversal (off-plane or vertical) and the lateral (in-plane), depending on the direction in which the heat is transported, relative to the surface of the device. In this work, the lateral heat flow is addressed, due to its easier fabrication process and compliance with the planar technology [16]. Fig. 1 shows the process flow used for fabrication of the TE converters.

A thin-layer of metal (aluminium – Al) is deposited by a direct current (DC) sputtering on a polyimide substrate. Then, the patterning of the metal layer is done, and the contacts are obtained (a). Next, the n-type Bi₂Te₃ thin-film is deposited by thermal coevaporation (b). The next step starts with the deposition of a negative photoresist (PR) layer, followed by an expose to UV (ultraviolet) light with a mask made of glass and nickel (to block the UV light) placed between the PR and the UV light source. After the exposition to the UV light, the UV-protected areas will be removed during the PR development (c). The n-type elements are patterned by photolithography (d). The n-type thin-film is etched

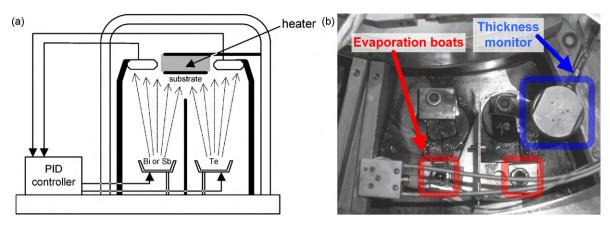


Fig. 2. For the system used in the co-evaporation. (a) A schematic of the complete system, and (b) a photography showing the inside of the co-evaporation chamber (the two evaporation boats and one of the thickness monitors are visible).

in HNO $_3$ and the p-type Sb $_2$ Te $_3$ thin-film is deposited by thermal co-evaporation (e). A new layer of PR is deposited, exposed to a UV light source and developed (f). The TE layer made of Sb $_2$ Te $_3$ is patterned by wet etching in a HNO $_3$:HCl bath (g) and finally, the PR is removed (h). A protective layer of silicon nitride (Si $_3$ N $_4$) can also be deposited by low-temperature hotwire chemical vapour deposition (HW-CVD) and patterned if required by the application.

2.1. Deposition of thin-films

TE thin-films were fabricated by thermal co-evaporation (see Fig. 2) in a high-vacuum chamber (with a base pressure of $\sim\!1.33\times10^{-4}\,\mathrm{Pa}$). Two large molybdenum boats (baffled boxes, with a volume of $4\,\mathrm{cm}^3$) were used at the same time, one for each of the elementary materials required to produce the desired compound. The power applied to each boat is controlled independently, using two computed proportional-integral derivative (PID) controllers [17] to maintain the deposition rate at user-defined constant values, during the deposition process. Two thickness monitors (quartz crystal oscillators) are carefully placed inside the chamber in such a way that each of them receives material only from the boat it is monitoring. A metal sheet is placed between the two boats to ensure deposition of a material at the respective quartz crystal

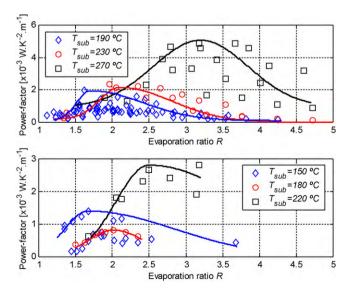


Fig. 3. Power-factor of Bi₂Te₃ (top) and Sb₂Te₃ (bottom) thin-films as a function of the Te/Bi evaporation flow rate ratio, *R*, and respective curve fittings (solid lines).

sensor only. The substrates are heated to the temperature set point (T_{sub}) in the range 150–270 °C.

The graphs showing the influence of the evaporation rate, R, of each material on TE properties of the compounds are presented in Fig. 3. The evaporation flow rate ratio, $R = F_{rTe}/F_{rBi,Sb}$, is defined as the amount (in volume of the deposited film) of tellurium (Te) divided by the amount of bismuth (Bi) – or antimony (Sb) – that arrives the substrate during deposition. The highest power-factor, PF, was obtained with a Bi (or Sb) evaporation rate of 2 Å s^{-1} and a Te evaporation rate of $6-7 \text{ Å s}^{-1}$, which corresponds to an evaporation flow rate ratio in the range 3-3.5.

The best values of $T_{\rm sub}$ for Bi₂Te₃ and for Sb₂Te₃ thin-films were about 270 and 220 °C, respectively. Finally, it must be noted that all thin-films were deposited on a polyimide (kapton) foil with a thickness of 25 μ m. Fig. 4 shows two SEM cross-section and surface images of both Bi₂Te₃ and Sb₂Te₃ thin-films, where their polycrystalline structure can be confirmed. Also, the former temperatures and the optimal evaporation flow rate ratios, R, were those which resulted in thin-films with larger grain size. This is of major concern, because a crystalline structure with an increased grain size is less resistive, whose consequence is a thin-film with an increased TE figures-of-merit, ZT.

2.2. Patterning

TE Bi_2Te_3 and Sb_2Te_3 thin-films (1 μ m thick) were deposited on the kapton substrate. The Transene's PKP negative photoresist was applied on the surface, and test structures were patterned by wet etching in the HNO3:HCl:H2O etchant (pure HNO3 and 37% HCl diluted in water). Fig. 5 shows the influence of the etchant composition on the etch rates. It was observed that an higher per cent of HCl (%HCl/%HNO3 > 0.5) induced cracking of the thin-film, whose consequence is the occurrence of peeling. Fig. 6 shows the influence of etchant dilution (in water) on the etch rate of Bi_2Te_3 and Sb_2Te_3 thin-films. For the case of Bi_2Te_3 thin-films, a dilution above 85% causes the occurrence of peeling. For dilutions below 65%, the etching occurred in a too fast manner and the end of the process was difficult to detect. The best results were obtained with the etchant composition in the range of 10:0:25 to 10:5:40 HNO3:HCl:H2O.

For the case of Sb_2Te_3 thin-films, the cracking and peeling of the thin-film occurred when a high percentage of HCl was presented in the etchant (%HCl/%HNO₃ > 0.5), as it was observed for the Bi_2Te_3 thin-films. The etch rate of the Sb_2Te_3 thin-films in diluted HNO₃ was about 50 times smaller than the etch rate of Bi_2Te_3 thin-films in the same etchant. This is important in terms of the selectivity of the process in the presence of both materials. The best results were obtained with the etchant of composition in the range 10:1:20 to 10:6:40 HNO₃:HCl:H₂O.

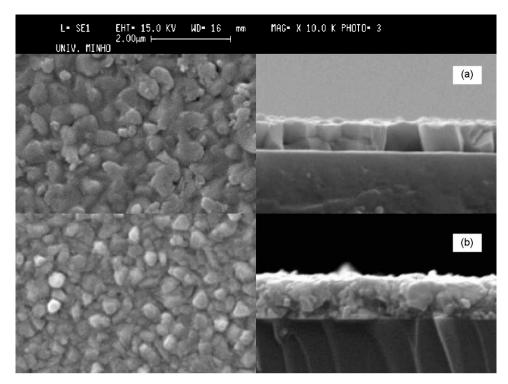


Fig. 4. SEM top view (left) and cross-sectional (right) images of Bi₂Te₃ (top) Sb₂Te₃ (bottom) thin-films.

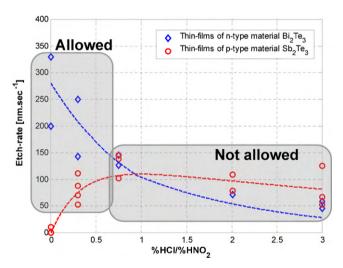


Fig. 5. Etch rate of Bi_2Te_3 and Sb_2Te_3 thin-films in $(1-x)HNO_3$:(x)HCl solution (diluted 70% in water, in volume).

The same solutions for tellurium compounds applied on thinfilms made of aluminium resulted in etch rates below $0.2\,\mathrm{nm\,s^{-1}}$ regardless of the etchant composition. Also, both $\mathrm{Bi_2Te_3}$ and $\mathrm{Sb_2Te_3}$ were slightly etched (<2 nm s⁻¹) by an aluminium etchant (16:1:1:2 phosphoric acid, nitric acid, acetic acid and water). Table 1 presents all relevant etch rates.

Table 1 Summary of etch rates.

Etchant	Material				
	Bi ₂ Te ₃	Sb ₂ Te ₃	Aluminium	Nickel	
Al-Transene type A 3HNO ₃ :HCl (dilution 70% H ₂ O)	$\begin{array}{c} 8\mathring{A}s^{-1} \\ 2000\mathring{A}s^{-1} \end{array}$	$5\text{\AA}\text{s}^{-1}$ $800\text{Å}\text{s}^{-1}$	$10-80 \text{Å} \text{s}^{-1}$ < $2 \text{Å} \text{s}^{-1}$	$<0.1 \text{Å} \text{s}^{-1}$ $<0.2 \text{Å} \text{s}^{-1}$	
H_2O) HNO_3 (dilution 70% H_2O)	$2500{\rm \AA}{\rm s}^{-1}$	$50\text{\AA}\text{s}^{-1}$	$< 0.1 \text{Å} \text{s}^{-1}$	$< 0.1 \text{Å} \text{s}^{-1}$	

Fig. 7 shows a planar TE microconverter, which was fabricated on top of a 25 μm thickness kapton foil. This microconverter is composed by eight pairs of TE elements and was fabricated with bottom contacts.

3. Experimental results

The in-plane thin-film electrical resistance was measured using the conventional four probe van der Pauw method, at the room temperature. The thermal conductivity (W m $^{-1}$ K $^{-1}$) was measured using the method proposed by Völklein [14], and the values of 1.3 and 1.8 W m $^{-1}$ K $^{-1}$ were obtained for the Bi₂Te₃ and Sb₂Te₃ thin-films (which were deposited as conditions to obtain the maximum power-factor), respectively. The measurements of the Seebeck coefficient were made by connecting one side of the thin-film to

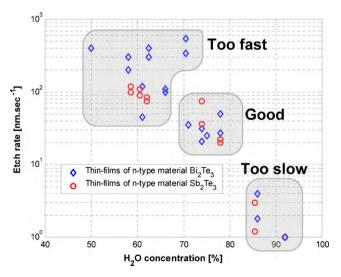


Fig. 6. Etch rate of Bi_2Te_3 and Sb_2Te_3 thin-films in 10:3 HNO₃:HCl solution, as a function of dilution in water (in volume).

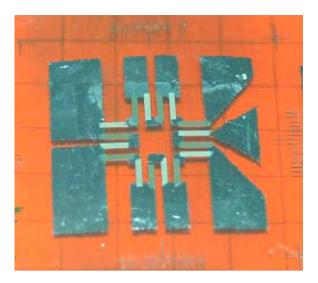


Fig. 7. A photography of a TE microconverter with eight pairs of TE elements, fabricated with bottom contacts.

a fixed temperature (heated metal block) and the other side to a heat sink at room temperature.

Fig. 8 shows a X-ray diffraction (XRD) spectrum of an optimised Bi_2Te_3 thin-film that reveals its polycrystalline structure. The peaks agree with the diffractograms for polycrystalline Bi_2Te_3 .

Fig. 9 shows a typical X-ray diffraction spectrum of an optimised thin-film that also reveals its polycrystalline structure. The peaks also agree with the power diffraction spectra for polycrystalline Sb_2Te_3 .

Tables 2 and 3 show the results of these measurements in the selected samples of Bi₂Te₃ and Sb₂Te₃ thin-films, as well as the corresponding figures-of-merit, *ZT*. These two following tables also list the *PF* for the selected samples of Bi₂Te₃ and Sb₂Te₃.

The measurements done in the selected samples showed an absolute value of the Seebeck coefficient in the range of 91–248 $\mu V\,K^{-1}$. An in-plane electrical resistivity of 7.6–39.1 $\mu \Omega\,m$ was obtained. The measurements for the Bi_2Te_3 and Sb_2Te_3 thin-films also revealed figures-of-merit, ZT, at the room temperatures of 0.97 and 0.56, and power-factors, PF, of 4.87 \times 10 $^{-3}$ and $2.81\times10^{-3}\,W\,K^{-1}\,m^{-2}$, respectively.

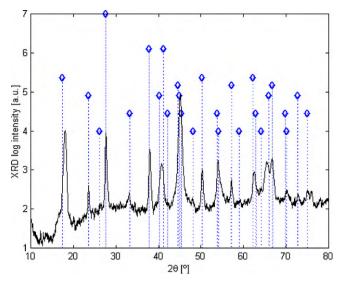


Fig. 8. For a sample of a n-type Bi₂Te₃ thin-film XRD analysis. The peaks agree with the power diffraction spectrum for Bi₂Te₃ (doted lines and diamonds).

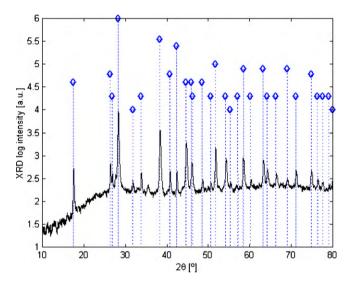


Fig. 9. For a sample of a p-type Sb_2Te_3 thin-film XRD analysis. The peaks agree with the powder diffraction spectrum for Sb_2Te_3 (doted lines and diamonds).

Table 2 Properties of the selected Bi₂Te₃ thin-films.

TF	T_{sub} (°C)	$R = F_{rTe}/F_{rBi/Sb}$	%Te by EDX	$\alpha (\mu V K^{-1})$	$\rho \left(\mu \Omega m \right)$	PF (W K $^{-2}$ m $^{-1}$)	ZT @ 300 K
#1	190	1.70	_	-180	16.6	1.95×10^{-3}	0.39
#2	230	2.10	62.8	-156	11.3	2.16×10^{-3}	0.43
#3		3.10	62.2	-152	13.4	1.72×10^{-3}	0.34
#4	240	3.20	59.1	-180	16.6	1.95×10^{-3}	0.40
#5	270	3.20	62.0	-248	12.6	4.87×10^{-3}	0.97
#6		3.90	-	-220	10.6	4.57×10^{-3}	0.91

Table 3 Properties of the selected Sb₂Te₃ thin-films.

TF	T _{sub} (°C)	$R = F_{rTe}/F_{rBi/Sb}$	%Te by EDX	$\alpha (\mu V K^{-1})$	$\rho \left(\mu \Omega \mathbf{m} \right)$	PF (W K $^{-2}$ m $^{-1}$)	ZT @ 300 K
#1	150	1.47	54.5	91	7.6	1.09×10^{-3}	0.22
#2		1.67	61.4	140	14.0	1.40×10^{-3}	0.28
#3	180	2.02	59.1	158	30.3	0.82×10^{-3}	0.16
#4		2.35	62.4	156	39.1	0.62×10^{-3}	0.12
#5	220	2.50	67.3	156	9.2	2.66×10^{-3}	0.53
#6		3.18	73.5	188	12.6	2.81×10^{-3}	0.56

4 Conclusions

This paper presented an IC-compatible fabrication technology for thermoelectric (TE) microdevices with high figure-of-merit, ZT. The deposition of thin-film Bi₂Te₃ and Sb₂Te₃ materials was done by co-evaporation on kapton substrates. The substrates were submitted to temperatures in the 190-270 °C range in experiments performed to find the best deposition conditions for the Bi₂Te₃ thin-films. The substrates also were submitted to temperatures in the range 150-220 °C for thin-films made of Sb₂Te₃. This paper also reported the influence of the deposition parameters on the TE properties of the thin-films. The thin-films were patterned by wet etching in HNO3:HCl:H2O and the influence of the etchant composition in the etch rate and pattern quality was measured. The optimised wet-etching results (i.e. an etch rate of $100-200 \,\mathrm{nm}\,\mathrm{s}^{-1}$ with high selectivity) were obtained with 10:3:30 HNO₃:HCl:H₂O. An etch rate below 0.2 nm s⁻¹ was observed in aluminium thinfilms, allowing a selectivity higher than 1000. The Bi₂Te₃ can also be etched in the 30% HNO₃ etchant, with a selectivity higher than 50 as compared to Sb₂Te₃ thin-films. The wet etching used is an advantage of the proposed fabrication technology, because of the cost advantage when compared with the reactive ion etching (RIE). Moreover, the etching does not impose limits on substrate temperature during the deposition. The kapton substrate can be bonded to a silicon substrate in a final process step. The IC-compatibility allows the integration with circuits and makes it an enabling technology for the realisation of self-powered wearable microsystems, where the temperature difference between the body temperature and ambient temperature provides sufficient energy for scavenging [18].

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Biographies



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