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REVIEW ARTICLE

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Fabrication strategies of flexible light sources based on micro/nano III-nitride LEDs

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ABSTRACT

The development of flexible optoelectronic devices has led to the appearance of new applications, ranging from wearable displays to medical implants. Hence, strategies have been developed to make flexible every component of the devices, including the light-emitting part. One such approach relies on the use of micro- or nano-light emitting diodes (LEDs) for their reduced footprint, allowing them to be easily separated from their substrate and embedded in a flexible matrix. In this review, the authors provide a comparison between the different geometries obtained by the growth of III-nitride structures for the fabrication of flexible devices. The processes used for their fabrication are then presented in detail. Last, an overview of the state of the art regarding flexible nanowire-based LED is provided, as well as some perspectives regarding their improvement.

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Nitrides; microLEDs; nanowire; flexible light source

1. Introduction

Flexible light sources and displays are emerging technologies poised for substantial market growth. Their applications range from curvilinear extended light sources, sometimes undergoing shape transformations, integrated into architectural design, to exploratory uses in biomedicine, especially for biosensors and micro-sized surgery robots, offering an advantage with their ability to conform to curvilinear surfaces. Compared to their traditional rigid counterparts, flexible displays and light sources stand out for their lightweight nature and increased resistance to shock hazards. While organic light-emitting diodes have made significant strides, demonstrating their suitability for mechanically flexible devices, issues like thermal instability, limited lifetime, and differential aging persist. In the last decade, many studies have described a variety of methods for the fabrication of flexible devices based on different inorganic materials including III-nitride semiconductors. Indeed, while III-nitrides are difficult to process into a flexible device due to their wurtzite crystalline structure, they have a high-power output [1], high efficiency [2], as well as resilience to thermal [3] and chemical [4] damages. Advanced strategies have been elaborated to fabricate flexible devices from these materials, both in the fabrication process and in the choice of the structures themselves as will be presented in this study. First, the authors aim to show why the downscaling of the structures toward micro- and nano-LED-based devices is an inherent advantage to the properties of the device. Second, the methods used to produce flexible devices made from different geometries are presented. Last, some insights are given into the perspectives of nanowire-based flexible emitters.

2. From planar flexible device toward µLED-based flexible device

2.1. Planar devices and issues

The mechanical properties of III-nitrides are not suitable for their use in flexible devices. Indeed, GaN is a stiff material, with a Young's modulus and yield strength of 295 and 15 GPa respectively [5] which results in plastic deformation for biaxial deformations larger than 5%. [5] More precisely, the bending stiffness [6] can be described with the following formula (in the case of a homogeneous thin film) [7]:

$$S = E \cdot t^3/12 \quad (1)$$

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with S the bending stiffness, E the young modulus of the thin film and t its thickness. As can be seen, the bending stiffness is a cubic function of the layer thickness. Hence, reducing the active layer thickness can greatly reduce the occurrence of plastic deformation and failure in the material. For example, Yoo *et al.* [8] demonstrated that relatively large ($100 \times 100\ \mu m^2$) mini-LEDs can be bent without damage. They performed a fatigue test consisting in 2000 bending cycles to a curvature radius of approx. 3.5 mm. The device did not suffer from any significant change of properties after the fatigue test as illustrated by highly reproducible current–voltage characteristics.

2.2. Advantages of µLEDs

Asad *et al.* [9] found that using smaller LEDs reduces the strain in the material for a similar bending radius (Figure 1) and concluded that an LED with a footprint smaller than 20 µm would experience a negligible strain for a 15 mm radius of curvature in their system (Figure 1(d–f)). In the case of µLEDs, the footprint of the structure can be reduced down to the µm scale [10]. Hence, considering that the aimed radius of curvature is usually of the order of several mm, the size reduction makes it possible to moderate or even completely avoid plastic deformation of the µLED material due to bending.

Apart from high tolerance to bending, µLEDs have numerous advantages, namely, they greatly increase the resolution of a display [11], they can be addressed individually [12] and have been demonstrated to be a viable option for monolithic RGB displays [13]. Thanks to these favorable properties, research on µLEDs and their application in rigid and flexible displays are constantly growing [14–16]. The reduced footprint of µLEDs facilitates their separation from the substrate enabling in some cases the use of a Mechanical Lift-Off transfer, which is not available with planar devices, that mostly rely on Laser Lift-Off or Chemical Lift-Off strategies. In the next section, the solutions available for the lift-off and subsequent transfer of nitride heterostructures will be presented. A summary of these methods along with their advantages and drawbacks are presented in Tables 1 and 2, respectively, for transfer and growth strategies.

3. Strategies for flexible device fabrication

The main approach for the fabrication of flexible devices relies on two steps: (i) the sample is encapsulated/bonded to a flexible material, and (ii) the heterostructure/polymer composite is separated from its substrate. The metal contacts that are used for the electrical injection of the device may be deposited before or after these two steps depending on what is the most

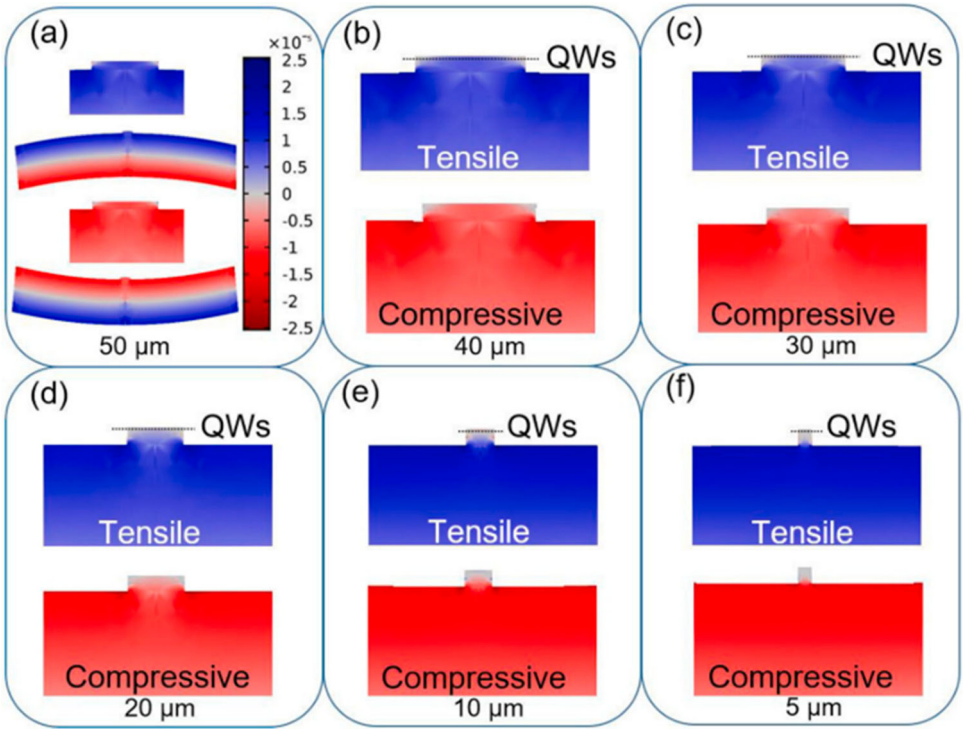

Figure 1. The simulated strain gradient for different sizes µLED on PI/Cu substrate with concave-down (Tensile stress) and concave-up (Compressive stress) bending radius of 15 mm; (a) 50 µm, (b) 40 µm, (c) 30 µm, (d) 20 µm, (e) 10 µm, and (f) 5 µm. The place of the quantum wells (QWs) is shown by a dashed line. (From ref. [9]).

Table 1. Summary of transfer methods, and assessment of their advantages and drawbacks.

Transfer Method	Advantage	Drawback
LLO	• Works independently of the geometry used • Reproducible	• Requires an expensive setup • Requires specific sbstrates
CLO	• Inexpensive • Reproducible	• Requires an appropriate choice of substrate/etchant • May induce pollution/contamination of the sample
Tape bonding / Scalpel assisted peel-off	• Inexpensive	• Requires strategies to reduce substrate/sample adhesion • Reproducibility issues • Difficult to automate
Transfer printing	• Reproducible (fully automated) • Adresses single LEDs	• Requires an expensive setup • Limits the minimal required size of LED

Table 2. Summary of the growth methods, and assessment of their advantages and drawbacks for flexible device fabrication.

Growth Method	Advantage	Drawback
Covalent epitaxy on a rigid substrate	• Mature technology • Wide choice of geometries and processes	• High sample/substrate adhesion • Growth defects due to lattice mismatch in covalent epitaxy
Van Der Waals/remote epitaxy	• Low sample/substrate adhesion • Strain relaxation at the interface	• Challenging growth • Technology is not mature
Epitaxy on flexible metal foil	• Doesn't require transfer	• Limits the available geometries for growth (growth on unusual substrates).

convenient in regard to the structure and separation technique used. The challenging part of this process is the separation of the heterostructure from the rigid crystalline substrate. In other semiconductors such as ZnO, this issue has been solved by using non-epitaxial low-temperature processes such as electrochemical deposition [17], which allows the direct deposition of the structures on flexible materials such as conductive polymer [18]. In the case of nitrides, the commonly used growth techniques are high-temperature epitaxial processes [19,20], which greatly limit the substrates that can be used for the growth. Hence, while not being the only solution, a major part of the flexible LED fabrication processes uses a transfer method. In the next paragraphs, we provide a list of strategies used to either facilitate or even to avoid the transfer step.

3.1. Laser lift-off

Laser Lift-Off (LLO) is a process that separates two materials by irradiating their interface with a laser [21]. This is done by choosing a specific wavelength for which one of the materials is highly transparent, and the other is highly absorbent. The laser beam goes through the transparent material and is absorbed at the interface, where it ablates the material. This process can be done on GaN grown on Sapphire since both materials have a large bandgap difference: 3.47 and 9.9 eV for GaN and Sapphire, respectively. Usually, the LLO is done using pulsed KrF lasers, with a wavelength of 248 nm and pulse durations of the order of the ns [21] or lower [22]. While the LLO was originally developed for planar structures [23–29] in order to break the high density of strong bonds between thin films and their substrates, it has also been used for μLED-based devices. [9,22,30,31] Still there are some drawbacks to this process (Table 1), namely, it is time- and energy-consuming and requires a specific apparatus that is expensive, and not versatile since it depends on the targeted material. However, because this method allows to process full-scale wafers and is reproducible, it appears to be a promising method for industrial-scale production. An example of device transfer on a flexible substrate using LLO is described in Figure 2. In this example, the samples are first adhered to a carrier substrate (Figure 2(a)), and subsequently separated from their growth substrate through LLO (Figure 2(b)). The obtained structures (Figure 2(c)) are subsequently contacted (Figure 2(d)) and transferred on a flexible substrate on which they can be operated (Figure 2(e-f)).

3.2. Chemical lift-off (CLO)

Chemical lift-off, as its name suggests, relies on the growth of GaN on a sacrificial material, which can be removed by wet etching. Some of the reports presented later on van der Waals growth on 2D materials also use this method, as the growth on a 2D material on SiO_2 allows to easily remove the sample from the substrate by buffered oxide etch [32,33]. A similar method can be applied to structures that are grown directly on Silicon substrates by wet etching [8,34]. Another possible method is to use a strongly doped n-GaN layer that can be electrochemically etched in acidic environments, which has already been used for the fabrication of flexible LEDs [35]. CLO is easy to implement, and relies on inexpensive products, but may introduce contaminants and damages to the heterostructure (Table 1). It also imposes limitations on the choice of the growth substrate, which should contain the sacrificial layer. However, as already said for the LLO method, this process holds promise for

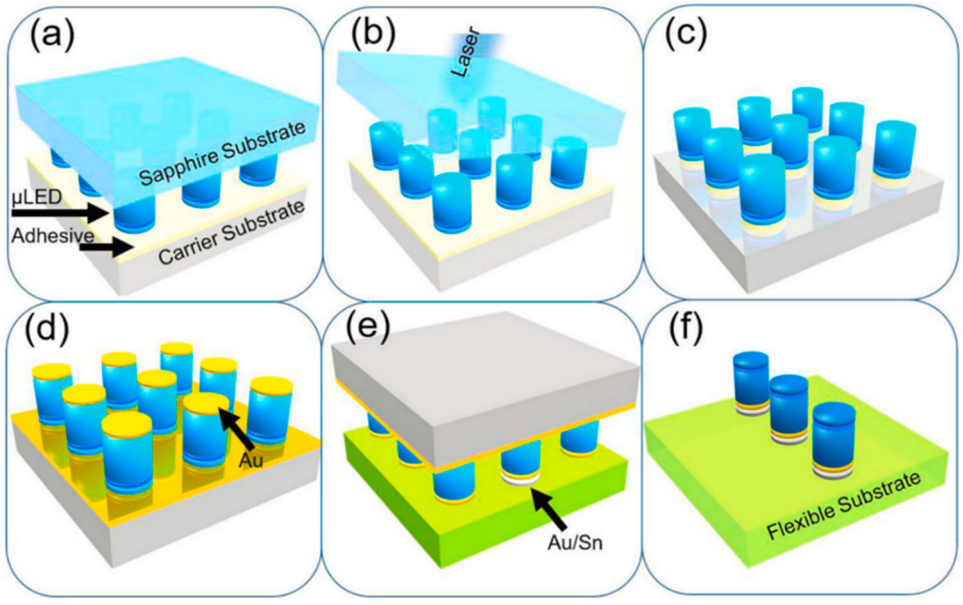

Figure 2. Example of an LED transfer using LLO. (a) The sample is bonded to a carrier substrate covered with an adhesive layer (a), the LLO is done to separate the Sapphire substrate. (c) The adhesive is dry etched, and a Cr/Cu/Au p-contact is deposited (d). The sample is then bonded to its final substrate by flip-chip bonding to an Au/Sn pad array, and the temporary carrier substrate is removed (f) (from Ref. [9]).

large-scale production, as it allows to process full wafers but does not require expensive apparatus to do so. An example of nanowire-based LED transfer using CLO is provided in Figure 3. In this example, the authors grew nitride nanowire LEDs on a graphene/SiO_2/Si substrate (Figure 3(a)), and subsequently encapsulated and contacted the nanowires. The flexible device is separated from its substrate by removing the SiO_2 in a buffered oxide etch bath. The membrane is then contacted on its n-side for operation. SEM pictures and device under operation can be seen in Figure 3(b,c).

3.3. Mechanical lift-off

Mechanical lift-off refers to any strategy that uses mechanical means to transfer the devices. It is not feasible to directly mechanically transfer an epitaxial planar heterostructure due to the strong bonding with the substrate. However, mechanical lift-off can be done if the adhesion of the heterostructure has been reduced, which can be achieved in several ways. One way to do so is to avoid classical covalent epitaxy, by growing on either an amorphous or a 2D material [36–40], but this is done at the cost of a significantly more complex growth process, as will be discussed in further detail in a following paragraph. The second option is to grow micro/nanostructured materials, with a small footprint and hence adhesion to the substrate [41]. Moreover, in the case of micro- or nano-wires, their high aspect ratio allows a torque to be applied at their base which facilitates further the lift-off [42–44]. Last, some studies report on the weakening of the GaN/substrate interface by the creation of voids [45].

There are several ways to mechanically separate the heterostructure from the sample. One possibility is to use a tape bonding process [36–40,45], which is mostly used in cases where the adhesion with the substrate was greatly reduced either through nanovoid formation or through growth on a 2D material. In this case, the heterostructure can be bonded by a tape, which can be removed from the substrate along with the heterostructure and released with heat [37,38], as shown in Figure 4(a), or with a mechanical force [36,39,40,45]. In a second method applied to nanowires embedded in a polymer matrix, a microscalpel blade is inserted between the polymer membrane and the substrate, which helps to break the nanowires at their bases and separates the membrane from the substrate [42,43], as presented in Figure 4(b). Both methods are cost-effective; however, they can create damage in the sample (Table 1). Moreover, they are usually done manually and are difficult to automate (except for Refs [37,38], which uses thermal release to separate the tape from the substrate). Hence, despite their promises for cheap processing, these methods still require some improvement in reproducible, as well as automation, to be suitable for industrial production. However, in the case of μLEDs, this last issue of lack of automation is addressed by the elastomer transfer-printing method. In this method, a stamp is used to pick up large areas of the array and transfer them to a flexible substrate (Figure 4(c)). In some cases, the release to

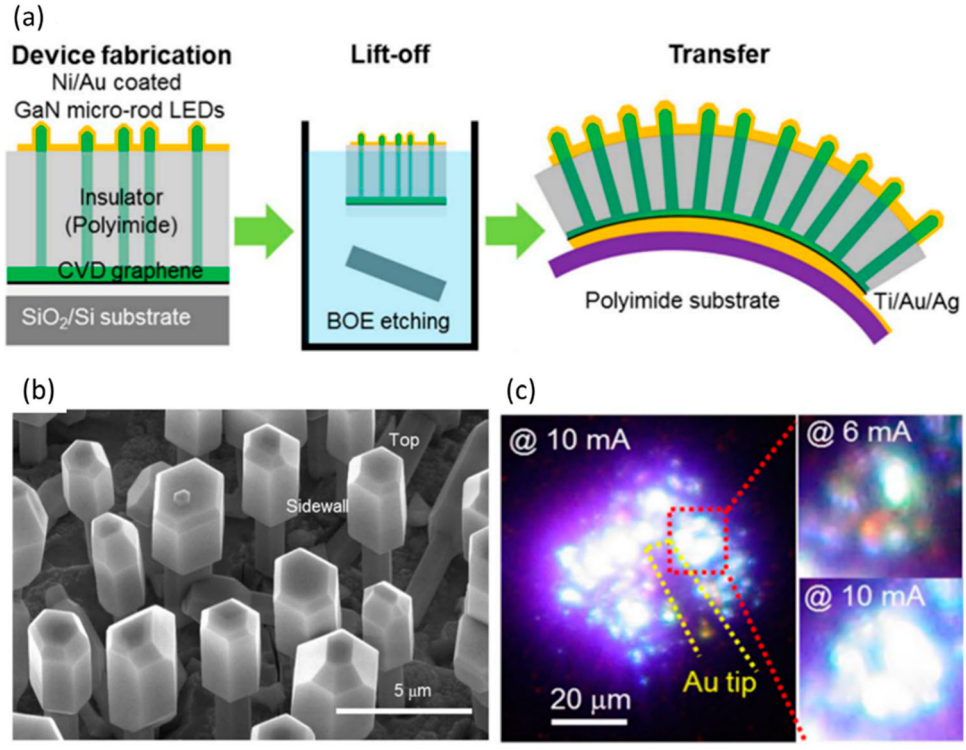

Figure 3. (a) example of a CLO transfer process, (b) corresponding SEM image of the sample, and (c) flexible LED under operation (from Ref. [32]).

the receiver substrate is laser-assisted [46]. This process can also be used to individually select and pick μLEDs, which allows to discard those who are dysfunctional [12]. Because this process can be automated, it addresses reproducibility issues (Table 1) and is already appropriate for the industrial processing of flexible LEDs. Additional methods are being developed as an alternative to stamping and include methods such as the electrostatic transfer [47,48] of the heterostructures.

3.4. Van Der Waals / remote epitaxy

Van Der Waals (VdW) / Remote Epitaxy are two processes in which the growth is done on a 2D material, e.g. like graphene [32,33,36–38] or h-BN [39,40]. Indeed, these materials lack dangling bonds, and contrary to standard substrates with sp^3 hybridization are not able to form any covalent bonds with the deposited material (Figure 5(a)). The growth results in an epitaxial material with a very low adhesion to its substrate, which facilitates the lift-off. The growth of a 2D material can occur through Van der Waals epitaxy, or through remote epitaxy. In the first case, the epitaxy occurs through weak Van der Waals bonding between the 2D material and the nitride layer, allowing for example to grow heterostructures on graphene transferred to amorphous materials such as SiO_2. In this case, there is no covalent bonding between the growing material and its substrate and the growth is driven by Van der Waals interactions originating from the dipole interactions between atoms. Van der Waals epitaxy does not require the use of additional crystalline structures beneath the 2D materials. For example, it allows for the selective area growth of GaN nanowires on patterned graphene on SiO_2/Si [49], showing that graphene imposes a crystallographic orientation to the GaN nanowires and yielding a good selectivity between the graphene and SiO_2. In the second case, the remote epitaxy is driven by the penetration of the polarization fields [50] of a crystalline material located beneath the 2D layer (such as for example SiC [51]) through the 2D material [52,53]. Hence, in this case, there is still an epitaxial relationship between the crystalline substrate underneath and the deposited material. This is the case in Ref. [54], in which the authors show the epitaxial relationship between GaN nanostructures on graphene and the underlying epitaxial SiC. In both cases, the growth occurs via low-energy bonds (instead of the covalent bonds present in classical epitaxy) owing to the small surface energy of the 2D materials. This leads to the growth of fully relaxed structures, that can easily be moved on their substrate or separated altogether, allowing for a wide range of transfer methods. This behaviour also means that the surface is antisurfactant, which complicates the material synthesis, and often promotes the growth of 3D

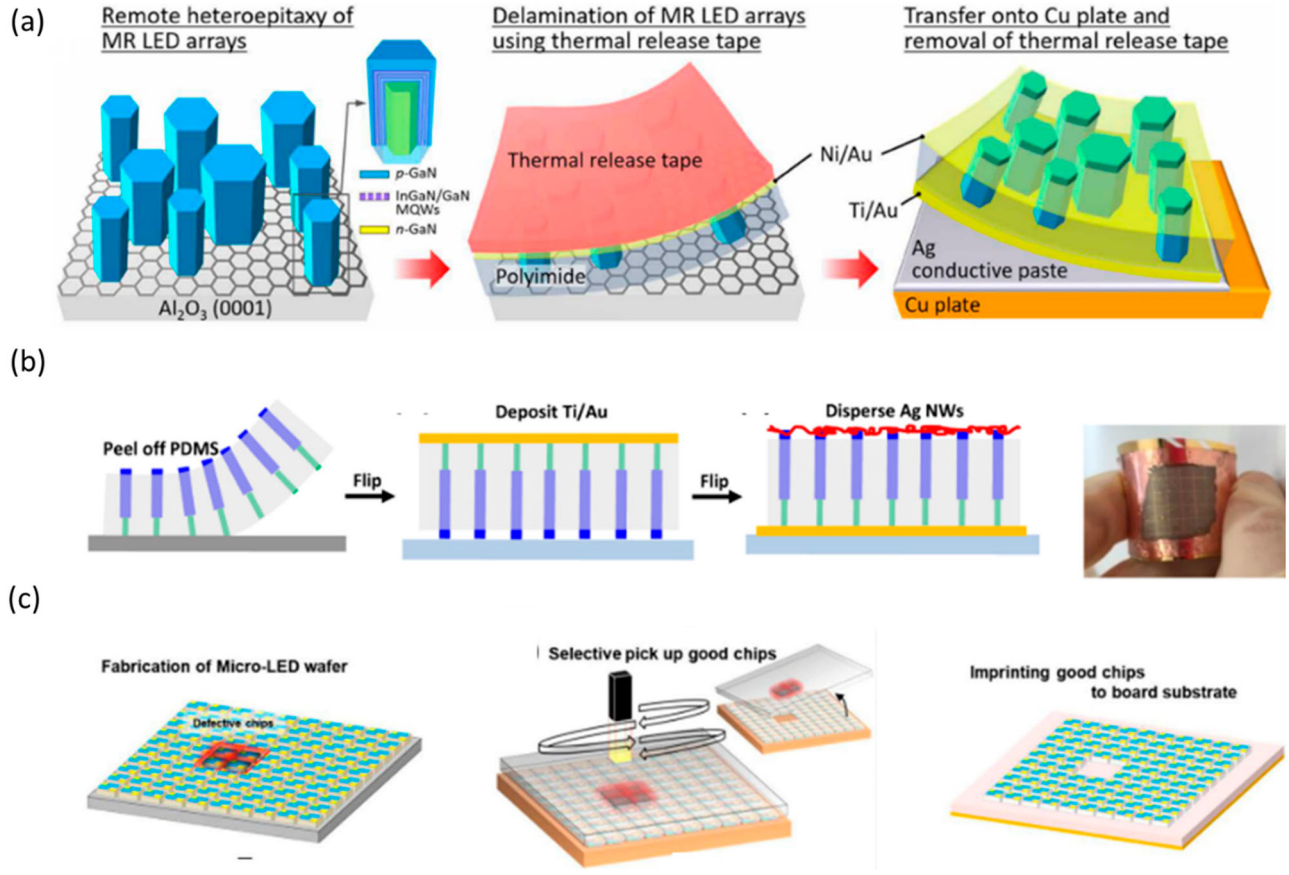

Figure 4. Methods for MLO lift off: (a) tape bonding MLO [37], (b) microscalpel peel off [42], (c) transfer printing for µLEDs [12].

structures within defects in the 2D layer instead of planar layers (Table 2). Although challenging, specifically for chemical vapor deposition processes [51], it is possible to grow a crystalline material on a 2D material, which demonstrated low adhesion to their surfaces. This method can be used [32,33,36–40] in combination with either CLO or MLO, as previously discussed (see Figure 3 and Figure 4(a)). Figure 5(b) and (c) schematically illustrate the nanowire growth on a graphene layer and the structural image of the interface. These wires were successfully processed into flexible LEDs which can stand a strong bending as shown in Figure 5(d). One additional advantage of the 2D materials is their potential use as a flexible electrode [29,55].

3.5. Growth on a metal foil

The use of flexible metallic foils for the epitaxy of GaN heterostructures has already been demonstrated in molecular beam epitaxy. In this method, the goal is to completely remove the transfer step. This process has been reported on Ta [56], Ti [56,57], Hf [58] and Mo [59] foils and leads to either nanorod structures, as seen in Figure 6(a), or to planar films [58,60]. The main advantage is that the method does not require extensive processes to transfer the heterostructure to a flexible carrier (Table 2). For example, Ref. [57] demonstrates the fabrication of a nanowire-based LED made by direct growth on Ta foils (Figure 6(a,b)), followed by a simple metal deposition for p-contact. Although the authors report contact issues leading to leakage and a decrease in intensity compared to the reference sample, the flexible device exhibits electroluminescence (Figure 6 (c)). This demonstration opens the way to the fabrication of flexible LEDs through a roll-to-roll process [60], which would be very efficient in the context of industrial production.

4. Advantages and drawbacks of nanowire-based flexible light emitters

Above we discussed the advantages of µLEDs over large-area planar LEDs for flexible device processing. Among the presented strategies, the nanowire geometry presents a few advantages for the fabrication of flexible devices [31–33,37,42–44,56,57,59,61]. First, the footprint of nanowires is reduced compared to classical µLED, ranging from a few µms [32,37,42,43,61] down to several hundreds of nanometres [31,33]. As stated previously,

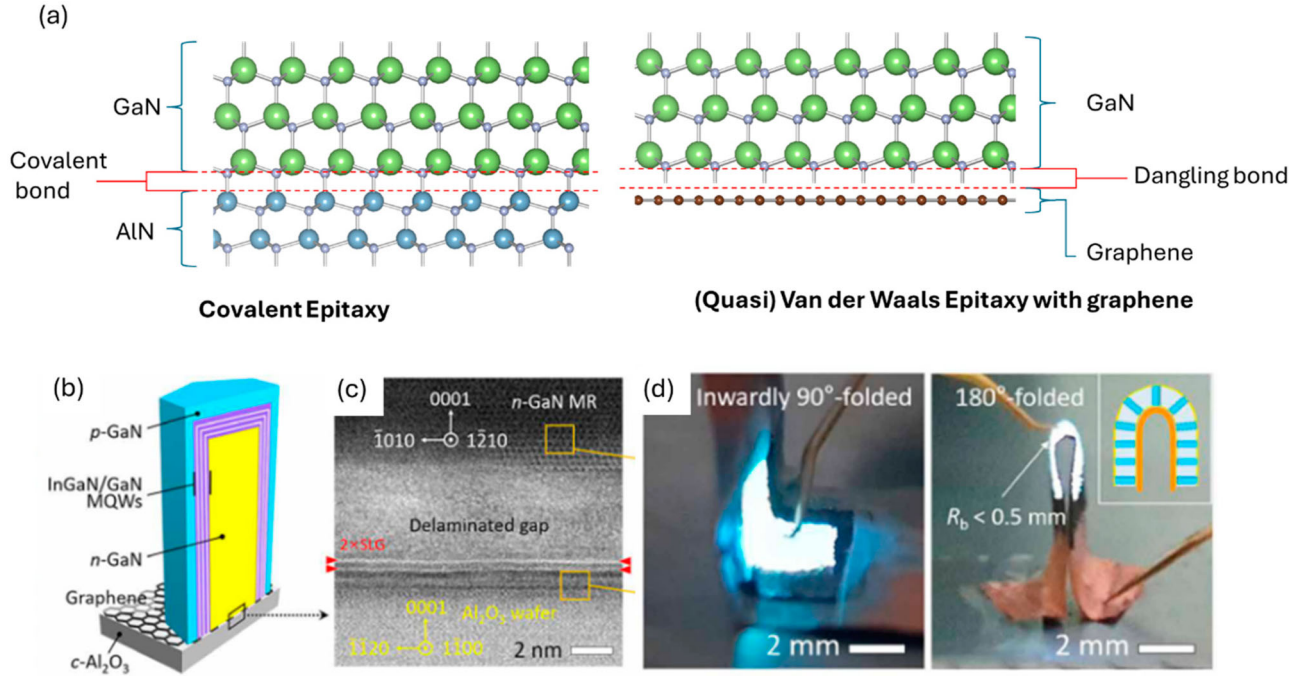

Figure 5. Schematic representation of boding in covalent and van der Waals epitaxy. (b) Scheme of a core-shell InGaN/GaN nanowire grown on graphene. (c) TEM Image of the interface between GaN/graphene/Sapphire. (d) corresponding device under operation. ((b), (c), and (d) from Ref. [37]).

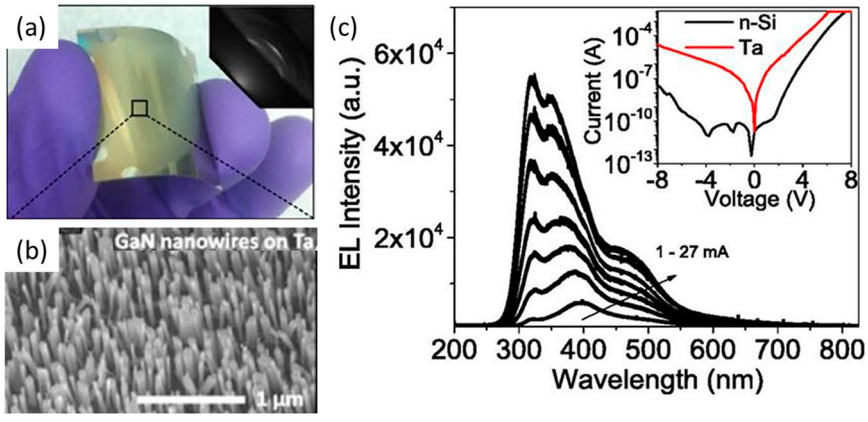

Figure 6. GaN nanowires grown on a flexible Ti substrate (from Ref. [56]). (a) Sample under bending, (b) high magnification SEM picture of the GaN nanowire array and (c) electroluminescence spectra of the device (I-V curves are shown in the inset for the flexible LED and for the reference sample on a Si substrate).

they can better withstand bending and can be more easily removed mechanically from the substrate. They have a high aspect ratio (with the height ranging from approx. 1 µm [31] to 100 µm), therefore in core/shell structures emitting from the sidewalls the total active area for optimized nanowire density can exceed the one of a planar device [41]. In addition, the In incorporation of nitride nanowires shows a strong dependence on the wire diameter [62], which makes possible the monolithic integration of full-color LEDs on a single chip [63]. Nanowire elaboration methods and their applications to emissive displays are reviewed in Refs [64,65].

To the best of our knowledge, the first reported growth of nanowire-based flexible devices using nitrides was published by Lee *et al.* [33], in which the authors used ZnO nanowires covered with an active shell made with InGaN/GaN. The structure was grown on graphene over SiO_2/SiO and encapsulated in a polyimide matrix before being transferred by CLO. Since then, other reports have been published using different substrates (Sapphire [31,42,43,61], metal foil [56,57,59], 2D materials [32,33,37], etc.) and various transfer methods (LLO [31], CLO [32,33], MLO, assisted [37] or not [42,43,61]). Despite not being the primary concern here, it is worth

noting that the devices made of encapsulated nanowires also can present some amount of stretchability [61].

A variety of nitride nanowire-based devices have been demonstrated, with different emissive properties. By using the tunability of the InGaN quantum well composition it is possible to obtain not only the standard blue but also the green emission from m-plane sidewalls of the nanostructures [66], as seen in Figure 7(a). Another method that has been used to tune the nanowire emission relies on the injection in different nanowire regions. Indeed, the emissive properties of various families of planes are different. In N-polar GaN nanowires, the semipolar r plane features a red-shifted InGaN emission, due to a higher In incorporation [42]. As a result, it is possible to modify the dominant emission by changing the operating voltage of the device, and selectively emit from the low energy c-plane InGaN QWs [42,67], as can be seen on the electroluminescence spectra in Figure 7(b).

The emission colour can also be modified by down-conversion using phosphors, which can be incorporated either as an additional layer on top of the device or in the encapsulating media itself. This approach allows to obtain white flexible NW-based LEDs [43] with the use of $Y_3Al_5O_{12}:Ce^{3+}$ nanophosphors as presented in Figure 7(c). By using mixtures of phosphors of various compositions, it is also possible to tune more finely the emission color to obtain a variety of white tones [68]. Another strategy to modify the emission of flexible nanowire LEDs is to use structures with dual emissions containing QWs of different composition, as presented in Ref. [69]. This allows in particular to get a monolithic white emission without requiring to use down-conversion by phosphors or other luminophores.

Despite the clear advantages of nanowire structures when it comes to the elaboration of flexible devices, their use is associated with some drawbacks. Due to the polymer encapsulation, flexible nanowire-based devices can be prone to overheating [70]. However, the Joule self-heating can be mitigated by using a dedicated heatsink, thus the device degradation can be avoided [70].

The biggest issue faced by nanowire devices is the light intensity inhomogeneity [32,33,37,42,43], as seen for example in Figure 8. The device is observed under an optical microscope without (Figure 8(b)) and with (Figure 8(c)) electrical injection. By superimposing both pictures (Figure 8(d)), one can notice that a significant number of nanowires are not producing electroluminescence, which results in inhomogeneities at macro-scale,

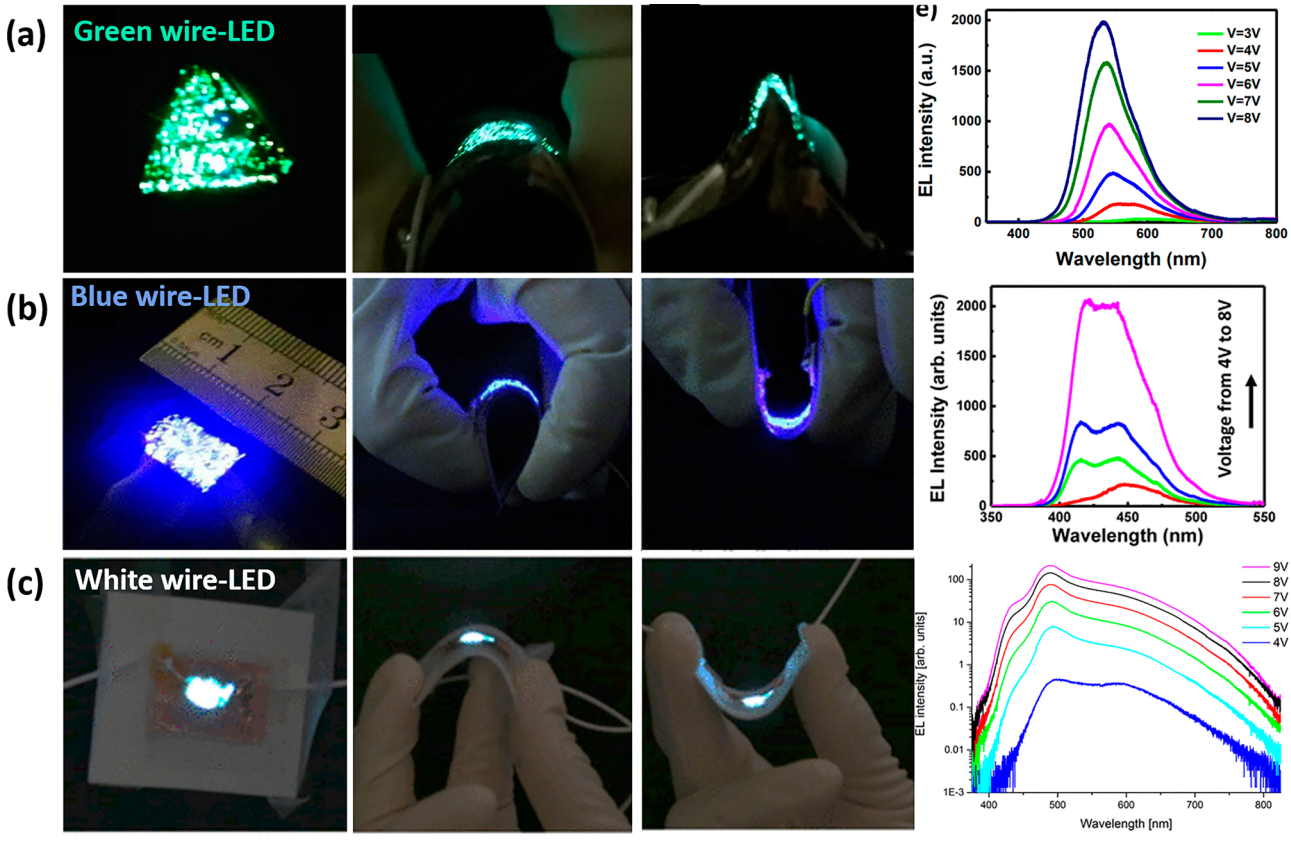

Figure 7. Demonstration of blue, green and white flexible LEDs based on core-shell nitride nanowires with and w/o positive/negative curvature and their corresponding emission spectra (from Refs [42,43]).

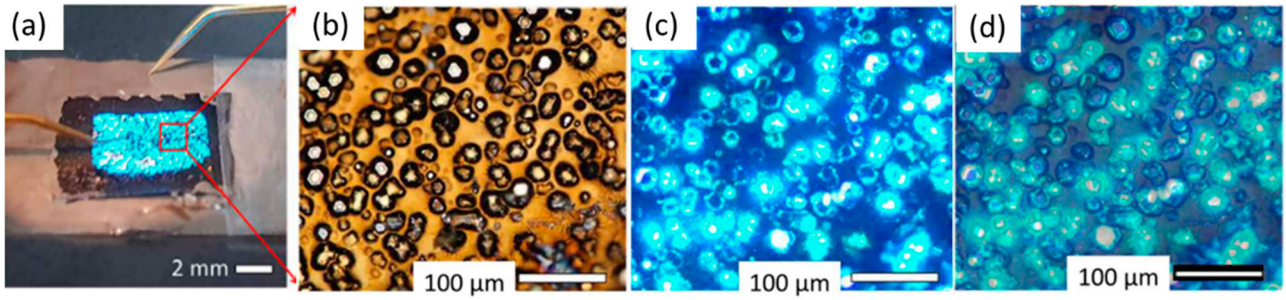

Figure 8. (a) operating nanowire-based flexible device. Sample (b) under an optical microscope, (c) under a microscope under injection, (d) combined picture of (a) and (b) (from Ref [37]).

as seen in Figure 8(a). This typically manifests as (i) an inhomogeneous intensity, leading to a spotty emission of the sample, and (ii) an inhomogeneous wavelength, which leads to parasitic contributions in the emission spectrum. The origin of these problems resides to a large extent in the use of a self-assembled nanowire growth [31–33,37,42–44,56,57,59,61]. Indeed, this process typically results in inhomogeneous geometries of the nanowires, leading to different lengths. Consequently, some of the nanowires are not long enough to extend through the membrane and be electrically injected [37]. Furthermore, due to different diameters and aspect ratios, the nanowires present different sidewall/top surface ratio and the In incorporation may also depend on the nanowire diameter leading to inhomogeneous overall emissions. Therefore, while the self-assembled process is advantageous for the proof-of-concept demonstration, the further optimization of flexible nanowire-based LEDs requires to use selective-area grown (SAG) nanowires. Flexible LEDs following SAG approach were successfully reported showing a good spectral stability under bending [31]. However, while SAG holds a great promise regarding the fabrication of nanowire-based flexible devices, it comes at the price of a significantly more complex and expensive process. Still this approach appears to be the only way to address issues linked with homogeneity and electrical injection faced by nanowire devices and despite the additional costs, the results presented by Aledia company in Ref. [71], demonstrate the usability of SAG nanowire structures for industrial devices such mid-size displays. Hence, the use of such structures should lead to a significant improvement of the properties of flexible light emitters, and their adoption in flexible displays.

5. Conclusions

In this paper, we have reviewed the present status of flexible LEDs based on inorganic micro- and nanostructures with a special focus on the fabrication methods. A variety of different approaches to produce flexible light-emitting devices have been reviewed, which include novel growth strategies using 2D materials, a specific design of the geometry of the structures and the choice of the dedicated transfer method. Regarding flexible devices elaborated from conventional thin films, µLEDs are shown to exhibit inherent advantages. Their geometry allows reducing the strain experienced by the active material during bending, making them appropriate candidates for the elaboration of flexible displays. The nanowire approach comes with promises of even higher device flexibility due to their tiny footprint which also facilitates their transfer, as well as of a strong luminance due to their extended area of emission. The flexibility of the nanowire LEDs can potentially be pushed to its extremes for emergence of new light sources allowing for twisting, stretching and folding. This extraordinary flexibility opens avenues for innovative applications, including wearable monitors, skin-applied medical devices, implantable light sources, and more. However, current nanowire devices suffer from homogeneity issues due to the growth process, and further work is required to obtain a nanowire-based fabrication process suitable for industrial production.

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