Photonic integration and fabrication technologies for on-chip active nano-devices in double tungstate gain materials

M. A. Sefunc, P.M. Muilwijk, R. T. Eachambadi, R.F. Russo, H.A.G.M. van Wolferen, G. Sengo, E.H. Bernhardi, M. Pollnau, and S.M. García-Blanco

University of Twente, Integrated Optical Micro Systems (IOMS) group, MESA+ Institute for Nanotechnology, 7500 AE Enschede, The Netherlands

Potassium double tungstates doped with different rare-earth (RE) ions, have been shown as promising materials to provide high, broadband, stable gain at different wavelengths including ~1 μm (Yb^{3+}), 1.55 μm (Er^{3+}) and ~2 μm (Tm^{3+}). In this paper, the utilization of this material in nanophotonic platforms will be presented. Several plasmonic structures of interest have been theoretically proposed. The integration and fabrication techniques required to produce these devices, namely bonding, thin layer transfer and focused ion beam milling have been developed. This work represents the first step towards the utilization of rare-earth doped double tungstates in nanophotonics.

Introduction

Potassium yttrium double tungstates, $KY(WO_4)_2$, (KYW) doped with different rare-earth (RE) ions, have been shown as promising materials to provide high, broadband, stable gain at different wavelengths including ~1 μm (Yb^{3+}), 1.55 μm (Er^{3+}) and ~2 μm (Tm^{3+}) [1]-[3]. A gain close to 1000 dB/cm has been recently demonstrated at a wavelength of 981 nm in Yb^{3+} doped KYW [1]. Such high gain makes this material a very interesting candidate for on-chip amplification in nanophotonic platforms. In particular, a promising application recently proposed theoretically, is the compensation of propagation losses in plasmonic waveguides and, in particular, long-range dielectric loaded surface plasmon polariton waveguides such as the ones depicted in Fig. 1 [4].
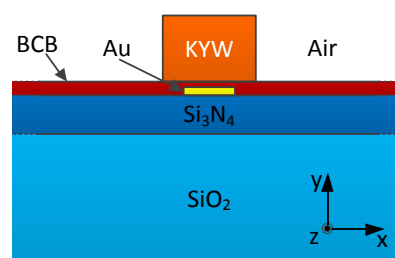

Figure. 1. Cross-section of the long-range dielectric loaded surface plasmon polariton structure proposed in [4] in which KYW was introduced as ridge material to compensate the propagation losses of the structure. The ridge dimensions for optimal loss compensation are 800 nm x 800 nm, the thickness of the Si_3N_4 layer is 350 nm, the thickness of the BCB layer is 100 nm and the thickness and width of the gold stripe are 15 nm and 200 nm respectively.

However, KYW is a crystalline material belonging to the monoclinic crystal family. In order to produce integrated devices, layers of rare-earth doped KYW are grown onto undoped substrates by liquid phase epitaxy [1]. In order to realize the structures

proposed in [4] (Fig. 1), a new set of technologies needs to be developed. In this work, the first preliminary results on bonding of KYW material to a silica-on-silicon (SiO$_2$-on-Si) platform, transfer of thin KYW layers by crystal-ion-slicing and patterning of the material with sub-micron precision by focused ion beam (FIB) are described.

Fabrication Techniques Required to Produce the Devices

Bonding

There are many methods available in the literature for bonding two layers together. For the structure presented in this paper, adhesive bonding with a thin layer of benzocyclobutene (BCB) appears as the most promising method. BCB (Cyclotene, code 3022-35, Dow Chemical) is optically transparent, has excellent planarization properties, a low curing temperature (250°C), does not form by-products during curing and has a high glass transition temperature (>350°C). Furthermore, BCB bonding has been proven to work with a great range of materials, including III-V materials, magneto-optic materials such as garnets, and lithium niobate [5]-[7].

BCB can be diluted with mesitylene to reduce its viscosity, resulting in thinner layers. Prior to bonding, both the SiO$_2$-on-Si and KYW samples are cleaned in an ultra-sonic bath using organic solvents (acetone and isopropanol) and deionized (DI) water. This is followed by a cleaning step in 99% HNO$_3$ clean. The samples are then dry-baked for 20 minutes at 150°C. Activation of the KYW surface is performed by an O$_2$-plasma cleaning step (20 minutes). The adhesion promoter AP3000 is spun on the SiO$_2$-on-Si substrate, followed by BCB (30 s at 3000 rpm). The BCB-coated SiO$_2$-on-Si substrate is then pre-cured for 4 minutes at 100°C.

The KYW sample and SiO$_2$-on-Si substrate are brought into contact at 150°C on a Finetech Fineplacer® Lambda bonder and kept for 20 minutes, while applying a pressure around 2-3 MPa. The BCB has its lowest viscosity, and thus the ability to reflow, at this temperature. The temperature is then ramped to 250°C, the pressure removed and the sample maintained for 1 hour at such temperature to complete curing of the BCB layer. After curing, the sample is cooled very slowly to room temperature. Slow cooling is required to prevent cracking of the KYW sample due to the mismatch of its coefficient of thermal expansion with that of the substrate. Bonding of KYW to SiO$_2$-on-Si has been shown to be possible [Fig. 2 (a)]. The bonding strength was sufficient to withstand a short lapping process from the 1 mm thickness of the bonded KYW sample to ~30 µm.

Thin Layer Crystalline KYW Transfer

A thin layer of a thickness ranging from 500 nm to 1 µm is necessary for the successful integration of KYW into the proposed structure (Fig. 1). The Smart-cut® method, also known as crystal-ion-slicing, has been successfully employed to transfer thin layers of many different crystalline materials [7]-[8]. In this method, the sample is first implanted with a high dose of energetic H$_2^+$ ions, followed by bonding, lift-off and subsequent fine polishing. The energy of the H$_2^+$ ions is chosen depending on the thickness required for the transferred layer.

A preliminary transfer of a partial thin layer of KYW was achieved using the ion-slicing method. A high dose (~8.3x10^{16} ion/cm^2) of H$_2^+$ ions with an energy of 180 keV was

utilized. Figure 2 (b) shows a SEM image of an area where the transfer of KYW was successful. Energy dispersive X-ray analysis (EDX) confirmed that the material transferred was KYW. Much process optimization is still needed to achieve the transfer of a full uniform thin layer. The effect of the ion dose, the temperature of the sample during implantation, the use of He^+ instead of H_2^+ ions and the temperature and time of the transfer process will be further investigated. However, this first result shows the good potential of the method to transfer thin layers of KYW.
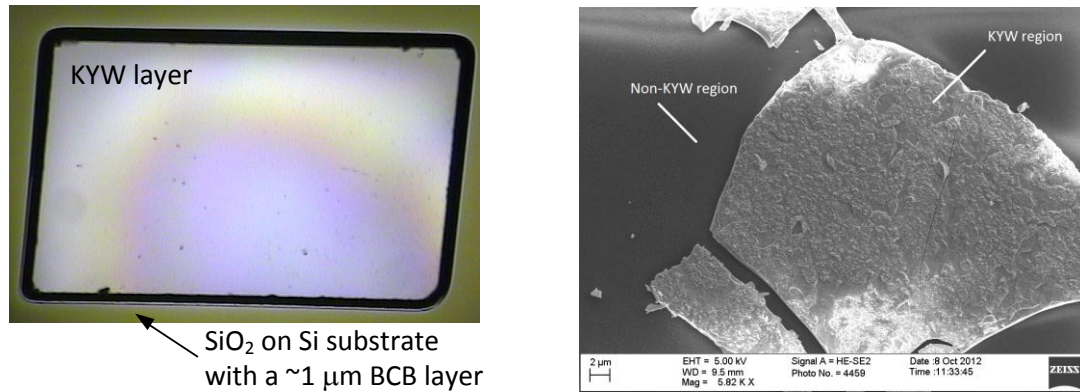

Figure 2: (a) Image of a small piece of KYW (3 mm x 4 mm) bonded to a silica-on-silicon substrate using BCB. Fringes indicate that there is some internal stress due to the bonding; (b) SEM image of KYW crystal over BCB coated silicon. Regions indicating the presence and absence of KYW are marked.

FIB (Focused Ion Beam) Milling

FIB milling is a commonly used fabrication technique for micro and nanofabrication. The accurate sectioning capabilities, its ability to etch complex patterns and the possibility to obtain high resolution images of the sample surface, make focused ion beam microscopes an ideal tool for milling a large variety of materials, including rare earth doped double tungstates.

In order to develop the process, an undoped KYW substrate with a ~3.5 μm layer of Yb^{3+} doped KYW grown on top was utilized. A 50 nm thick layer of titanium was deposited to avoid charging effects during milling. The dimensions of the channels, both cross section and length, are optimized using PhoeniX FieldDesigner (PhoeniX B.V.) considering the mode confinement in the channels and the propagation losses. The developed milling process consists of two steps. In the first step, "milling" is used to obtain the pattern. High voltage and ion beam current values utilized were 30 kV and 21 nA respectively. The second step consist of the polishing of the input and output channels. The FIB settings (voltage and ion beam current) for the polishing step were 30 kV and 21 nA for the front-end facets and 30 kV and 2.8 nA for the back-end facets. The scan direction for the polishing process was from bottom to top. A drift-compensation tool was used during the entire fabrication process. Figure 3 (a) shows an SEM picture of the milled samples. Figure 3 (b) shows a close up SEM of the surface roughness. The obtained results in terms of smoothness of sidewalls and end-facets, together with the almost total absence of re-deposition of materials and damage on the structures, make this fabrication method promising for milling the double tungstates gain materials.
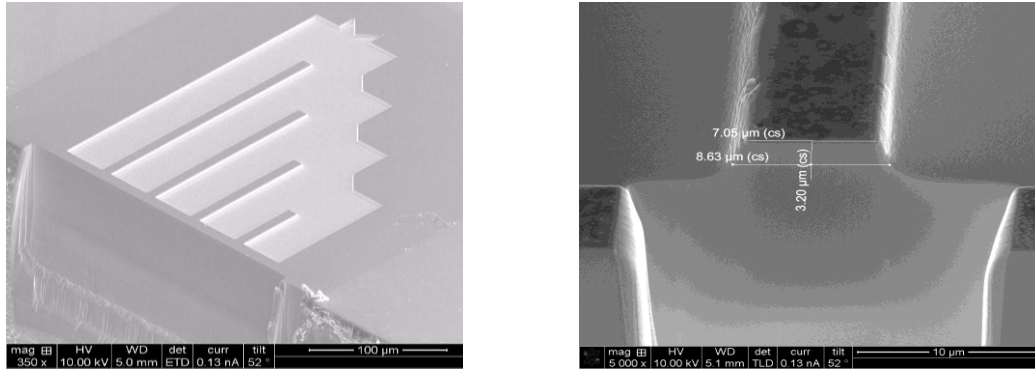
Figure 3: (a) Overall view of the structure fabricated using FIB: four channels (from right to left; 50,100,150,200 μm long respectively) and the anti-reflection corners. The picture is taken before the cleaning cross section process for the front-end facets; (b) Detail of the FIB polished end-facet. A low surface roughness of both the end-facets and side-walls can be observed.

Conclusions

Several plasmonic structures incorporating rare-earth ions doped potassium double tungstate gain material to provide gain and eventually to compensate the propagation losses produced by the metal layer have been previously proposed [4]. Integration and fabrication techniques such as bonding, thin layer transfer and FIB milling, have been investigated as potential technologies to fabricate the proposed structures. The fabrication steps considered in this paper show potential to utilize RE-ions doped potassium double tungstate materials in nanophotonic platforms. However, a great amount of process optimization is still required and is ongoing to arrive to efficient and reproducible processes that will permit to realize these devices.

References

[1] D. Geskus, S. Aravazhi, S. M. García-Blanco, and M. Pollnau, "Giant optical gain in a rare-earth-ion-doped microstructure," Adv. Mater., vol. 24, OP19-OP22, 2012.

[2] W. Bolaños, J. J. Carvajal, X. Mateos, M. C. Pujol, N. Thilmann, V. Pasiskevicius, G. Lifante, M. Aguiló, and F. Díaz, "Epitaxial layers of $KY_{1-x-y}Gd_xLu_y(WO_4)_2$ doped with Er^{3+} and Tm^{3+} for planar waveguide lasers," Opt. Mater. vol. 32, 469–474, 2010.

[3] K. van Dalfsen, S. Aravazhi, C. Grivas, S. M. García-Blanco, and M. Pollnau, "Thulium channel waveguide laser in a monoclinic double tungstate with 70% slope efficiency," Opt. Lett. Vol. 37, 887-889, 2012.

[4] García-Blanco, S. M., Pollnau, M., and Bozhevolnyi, S. I., "Loss compensation in long-range dielectric-loaded surface plasmon-polariton waveguides," Opt. Express, vol. 19, 25298-25311, 2011.

[5] S. Stankovic, R. Jones, J. Heck, M. Sysak, D. Van Thourhout, and G. Roelkens, "Die-to-Die adhesive bonding procedure for evanescently-coupled photonic devices," Electrochemical and Solid-State Letters, vol. 14, h326-h329, 2011.

[6] S. Ghosh, S. Keyvavinia, W. Van Roy, T. Mizumoto, G. Roelkens, and R. Baets, "Ce:YIG/Silicon-on-Insulator waveguide optical isolator realized by adhesive bonding," Opt. Express, vol. 20, 1839-1848, 2012.

[7] G. Poberaj, M. Koechlin, F. Sulser, A.a Guarino, J. Hajfler, and P. Günter, "Ion-sliced lithium niobate thin films for active photonic devices," Opt. Mater., vol. 31, 1054–1058, 2009.

[8] M. Bruel. "Application of hydrogen ion beams to silicon on insulator material technology," Nuclear Instruments and Methods in Physics Research, vol. 108, 313-319, 1996.