

# Tantalum-tungsten alloy photonic crystals for high-temperature energy conversion systems

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## ABSTRACT

A tantalum tungsten (Ta-W) solid solution alloy, Ta 3% W, based 2D photonic crystal (PhC) was designed and fabricated for high-temperature energy conversion applications. Metallic PhCs are promising as high performance selective thermal emitters for solid-state thermal-to-electricity energy conversion concepts including thermophotovoltaic (TPV) energy conversion, as well as highly selective solar absorbers/emitters for solar thermal and solar TPV applications due to the ability to tune their spectral properties and achieve highly selective emission. The mechanical and thermal stability of the substrate was characterized as well as the optical properties of the fabricated PhC. The Ta 3% W alloy presents advantages compared to the non-alloys as it combines the better high-temperature thermo-mechanical properties of W with the more compliant material properties of Ta, allowing for a direct system integration path of the PhC as selective emitter/absorber into a spectrum of energy conversion systems. Furthermore, the thermo-mechanical properties can be fine-tuned by the W content. A 2D PhC was designed to have high spectral selectivity matched to the bandgap of a TPV cell using numerical simulations and fabricated using standard semiconductor processes. The emittance of the Ta 3% W PhC was obtained from near-normal reflectance measurements at room temperature before and after annealing at 1200°C for 24h in vacuum with a protective coating of 40nm HfO<sub>2</sub>, showing high selectivity in agreement with simulations. SEM images of the cross section of the PhC prepared by FIB confirm the structural stability of the PhC after anneal, i.e. the coating effectively prevented structural degradation due to surface diffusion.

**Keywords:** Thermophotovoltaics, Photonic crystals, High-temperature energy conversion, Tantalum-tungsten alloy, Spectral selectivity, Thermal barrier coating, Selective emitter/absorber.

## 1. INTRODUCTION

An increased interest in thermophotovoltaics (TPV), solar TPV, and solar-thermal energy conversion systems in general has led to new investigations of photonic crystals (PhCs) as selective emitters/absorbers made from refractory metals that can maintain optical properties at high temperatures over operational lifetime. These two-dimensional (2D) PhCs are fabricated by etching a periodic pattern into a metallic substrate, such as tungsten (W)<sup>1-6</sup> or tantalum (Ta)<sup>7</sup> which are suitable substrates for PhCs for energy applications due to their high long-wavelength reflectance as well as high melting point and low vapor pressure. These metallic 2D PhCs can

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be designed to exhibit precisely tailored optical properties and a highly selective spectral emittance which is matched to the band gap of a suitable small band gap photovoltaic cell. This way, the PhCs produce spectrally confined selective emission of light at high operating temperatures ( $> 900^{\circ}\text{C}$ ).<sup>2,3,6-8</sup> High reflectance, *i.e.* low emittance, in the infrared (IR) wavelength range of the substrate material is critical for high-temperature nanophotonic devices, to reduce losses due to waste heat at wavelengths longer than the bandgap of a TPV cell (typically 2-3  $\mu\text{m}$ ). Similarly, for selective solar absorbers low emittance at long wavelengths minimizes re-radiation. In advanced TPV or solar TPV systems, selective absorbers/emitters have to be integrated into the system by welding onto the the heat source/sink. The heat source can be a micro-reactor fueled by hydrocarbon combustion,<sup>9</sup> a radio-isotope general purpose heat source (GPHS),<sup>10</sup> or thermal fluid in the case of a solar absorber.

A tantalum tungsten solid solution alloy, Ta 3% W, was investigated as a substrate for a 2D PhC for high-temperature high-efficiency TPV. The Ta 3% W alloy presents critical advantages compared to non-alloys as it combines better thermo-mechanical properties of W with the more compliant material properties of Ta, allowing a direct system integration path, *i.e.* machining and welding. In addition, the mechanical stability of the selective emitters is greatly enhanced at high temperature using Ta-W alloy substrates as opposed to pure Ta, which is beneficial for overall system stability where degradation such as creep and deflection can play a critical role in system failure. A 2D PhC on a pre-annealed polycrystalline Ta 3% W alloy substrate was designed using finite-difference time-domain (FDTD) numerical simulations<sup>11</sup> and fabricated using standard semiconductor processes.<sup>12</sup> The emittance of Ta 3% W was obtained from near-normal reflectance measurements at room temperature before and after annealing at  $1200^{\circ}\text{C}$  for 24 h in vacuum with a protective dielectric coating of 40 nm  $\text{HfO}_2$ . A slight degradation of the initial high spectral selectivity of the emittance spectrum after anneal was attributed to the beginning of carbide formation on the surface of the  $\text{HfO}_2$  coating, which is greatly decelerated in contrast to the surface reaction on Ta without the coating.<sup>13</sup> Scanning electron microscope (SEM) images of the cross section of the PhC prepared by focused ion beam milling (FIB) compared before and after anneal confirm the structural stability of the  $\text{HfO}_2$  coated Ta 3% W PhC. No degradation (*i.e.* rounding of the sharp features) was observed, which confirms that the coating effectively prevents structural degradation due to surface diffusion.

## 2. DESIGN AND FABRICATION

### 2.1 2D Photonic Crystal Design

In order to maximize the efficiency of a TPV system such as a micro-reactor fueled by hydrocarbon combustion,<sup>9</sup> a 2D PhC is welded directly on the source of heat. The emittance spectrum of the PhC is tailored so that the cutoff wavelength  $\lambda_{cutoff}$  of the high emittance band matches the bandgap  $\lambda_{PV}$  of a PV cell, such as a GaSb or InGaAsSb TPV cell. The thermal emission of previously fabricated Ta PhCs shows high selectivity as well as a marked emission peak below the target cutoff wavelength while emission at higher wavelengths  $\lambda \geq 3\mu\text{m}$  is kept low<sup>13</sup> (Fig. 1), which enables TPV systems to have unprecedented high efficiencies.

The PhC consists of a periodic array of etched cylindrical cavities with period  $a$ , radius  $r$ , and depth  $d$  in the substrate (Ta 3% W). The geometric parameters of the cavity array define the cutoff wavelength which is tuned by selecting the appropriate  $r$  and  $d$ . Prior to fabrication, the PhCs are simulated and the geometric parameters are optimized using a FDTD algorithm<sup>14</sup> implemented in Meep.<sup>11</sup> For the optimized set of parameters  $r$  and  $d$ , the quality (Q) factors of the radiative and absorptive modes of the PhC are matched so that the emittance below the cutoff wavelength is maximized.<sup>6,8</sup> At the same time, the emittance above the cutoff wavelength is kept low in order to minimize losses in the IR due to waste heat and to achieve high selectivity with a sharp

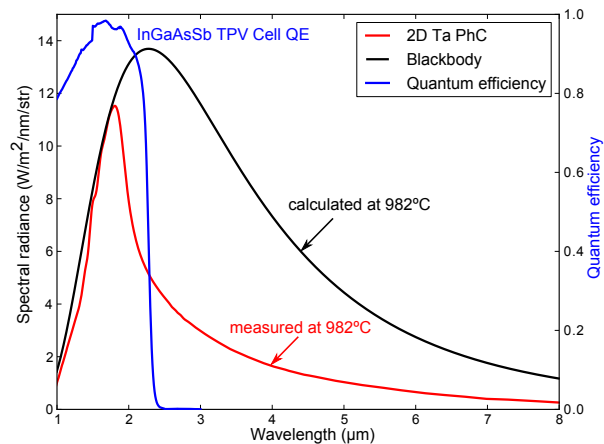


Figure 1. (color online) PhCs are designed to selectively emit in the region of interest, *i.e.* below  $\lambda_{PV}$ . Left axis: Comparison of measured normal spectral emission of a Ta PhC at 982°C with the calculated blackbody emission at 982°C. Right axis: Measured Quantum Efficiency (QE) of a InGaAsSb quaternary PV cell with a bandgap  $\lambda_{PV} = 2.3\mu\text{m}$ .

cutoff between the two regions. The optical dispersion of the substrate is taken into account in the simulation using a Drude-Lorentz model fit to the measured reflectance of polished Ta 3% W, which is almost identical to that of a pure Ta substrate.

## 2.2 Materials and Fabrication

The intended use of the selective emitters in high-temperature energy conversion applications with target operating temperatures  $> 900^\circ\text{C}$  and expected lifetimes of years imposes strict requirements on both the thermal stability of the fabricated microstructures and their optical properties as well as the thermal and thermo-mechanical stability of the emitter substrates in the context of system integration. Refractory metals are preferred at high temperatures due to their high melting point, low vapor pressure and advantageous high reflectivity in the IR. W has high yield strength and Young's Modulus however it is very brittle and therefore hard to machine or weld. Ta on the other hand is soft and more compliant, in addition to being easily weldable and machinable. In order however for a Ta based system to achieve the same mechanical stability as for a W based system, a thicker substrate is required due to Ta's softness, which in turn adds to the system weight and cost. The use of Ta-W alloys allows one to achieve the same thermo-mechanical stability while keeping the required material thickness low. The substrates used in this study were prepared from an alloy of high purity polycrystalline Ta and W (2 – 3.5% concentration) which we refer to as Ta 3% W in this manuscript. Fig. 2 illustrates the mechanical properties of Ta, Ta 3% W and Ta 10% W and their dependence on temperature and shows the increasing mechanical stability with increasing W content of the alloy for all temperatures.<sup>15</sup> The substrates were annealed in a Ta-lined high vacuum furnace at 1755°C for 8 h under high vacuum to achieve thermal stability of the polycrystalline substrate material with large grain size. The substrates were cut, lapped to a thickness of 1 mm, and chemo-mechanically polished to achieve a roughness  $R_a$  of less than 10 Å.

The PhC fabrication process for the Ta 3% W substrates is similar to the one developed for Ta and W substrates using interference lithography and reactive ion etching (RIE) of a hard mask and a subsequent deep RIE (DRIE) of Ta.<sup>5,6,12</sup> A 100 nm layer of Chrome (Cr) and 40 nm layer of SiO<sub>2</sub> were deposited by e-beam evaporation onto the cleaned substrates to be used as a hard mask for the Ta etching. An anti-reflection coating (ARC, AZ BARLi) of about 270 nm was then spin coated and another 10 nm layer of SiO<sub>2</sub> was e-beam evaporated onto the sample. The SiO<sub>2</sub> layer on top of the ARC serves as a protection layer when etching and ashing the ARC. Finally a layer of about 250 nm of negative photoresist (THMR-iNPS4, OHKA America) was spin coated onto the sample for the lithography process. Interference lithography (IL) is a relatively simple, inexpensive and precise method for maskless lithography with high resolution for large areas. Two coherent laser beams interfere

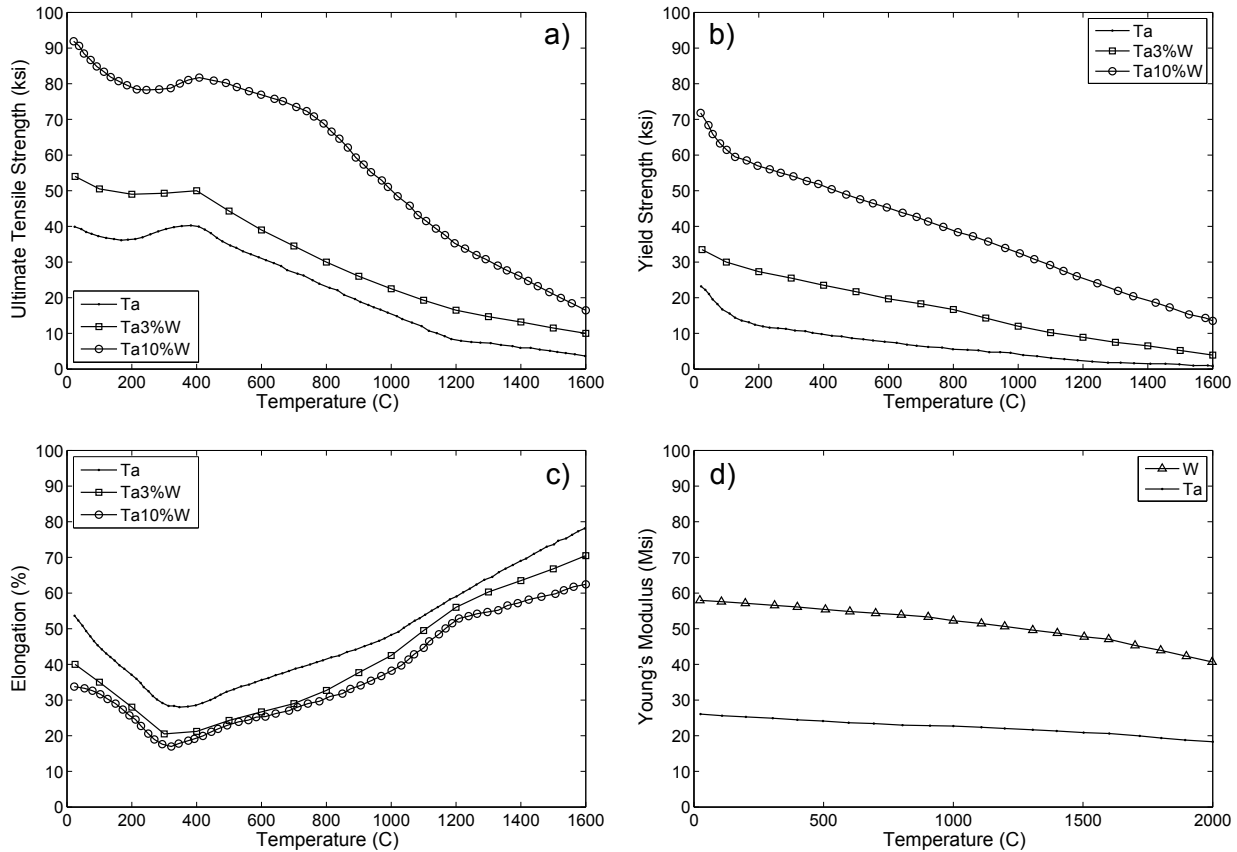


Figure 2. Comparison of (a) Ultimate Tensile Strength (UTS), (b) Yield Strength (YS) and (c) percent elongation of Ta, Ta 3% W and Ta 10% W and their dependence on temperature. (d) Comparison of Young's modulus of Ta and W.

with each other creating an interference pattern that is recorded in the photoresist by exposure, creating a 1D or 2D pattern. For small samples, a Lloyd's mirror (LM) setup<sup>16</sup> can be used for the exposure. For a larger periodic pattern with high uniformity, such as the one required for our substrates, IL is performed using a Mach-Zehnder (MZ) setup with a 325 nm HeCd Laser.<sup>17</sup> The periodicity of the pattern  $p$  is defined by the interference angle  $\theta$  as  $p = \lambda/2 \sin \theta$ . The exposure is performed once, and then once more after the samples are rotated by 90° to create a square array of cylindrical cavities. The target diameter of the cylindrical cavities for a cutoff wavelength of 2.0  $\mu\text{m}$  is 1.08  $\mu\text{m}$  and the target period 1.3  $\mu\text{m}$  based on simulations discussed in Section 3. Due to the Gaussian distribution of the intensity of the exposure beam, the diameter of the holes is not completely uniform across the sample. Since the exposure dose is decreasing with the distance from the center of the substrate, the hole diameter is increasing slightly towards the edges of the sample. The starting diameter after exposure is on the order of 500 nm (limited by the nature of IL) and has to be increased in order to achieve the target size in the Cr layer, which will then be used as a mask for etching the Ta 3% W substrate. The increase in hole size is achieved by isotropic plasma ashing of the photoresist and the ARC layers. After the photoresist is exposed using commercial CD-26, it is partially removed by O<sub>2</sub> plasma ashing. Longer ashing times result in larger hole diameters, however this is limited by the thickness of the resist. Generally an increase of the cavity diameter of about 200 nm is achieved by ashing the photoresist for 90 s at 200 W. The pattern is transferred by RIE into the SiO<sub>2</sub> and ARC layers using a CHF<sub>3</sub> and He/O<sub>2</sub> process respectively, in a Plasmatherm 790. Once the desired diameter is obtained by ashing the ARC (with a rate of approximately  $\Delta 2r = 2\text{nm/s}$  at 200 W) the cavities are etched into the underlying SiO<sub>2</sub> layer by the same CHF<sub>3</sub> based RIE process, and subsequently into the Cr layer by a Cl<sub>2</sub>/O<sub>2</sub> based RIE process using a Nexx Cirrus 150. It was observed that Ta 3% W can be etched

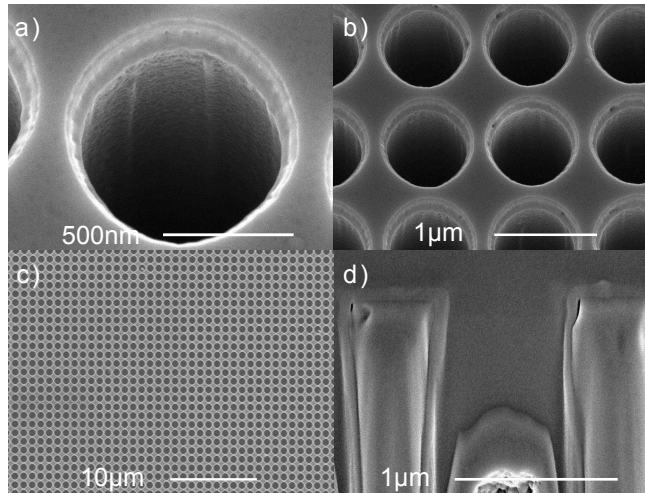


Figure 3. SEM top views of the Ta 3% W PhC demonstrating the high quality of the the RIE and DRIE. Previously observed grass effects are no longer present.

in the same way as pure Ta. The final pattern transfer into the Ta 3% W substrate is done by DRIE (Alcatel AMS100) with a Bosch process using  $\text{SF}_6$  (200 sccm) and  $\text{C}_4\text{F}_8$  (100 sccm) with 3 s/1.5 s pulses as the etching and passivating species, respectively, power 1200 W, pressure  $\sim 3$  mTorr, substrate bias 75 W, and temperature  $20^\circ\text{C}$ . The pulse duration was optimized to achieve complete passivation of the sidewalls and a straight sidewall etch profile, and the etch time was limited to 5 min cycles to prevent overheating of the substrate. An etch depth of about  $6\ \mu\text{m}$  corresponding to an aspect ratio of about 6 was achieved in an overall etch time of 10 min. After DRIE of Ta 3% W, the remaining Cr layer is removed by Cr-7 (Cyantek) liquid etchant.

Surface diffusion and surface chemical reactions are known high-temperature PhC degradation mechanisms. Previous studies have shown that the addition of a protective dielectric coating such as  $\text{HfO}_2$  onto the fabricated selective emitters prevented both structural degradation by surface diffusion and grain boundary grooving as well as surface reactions such as the formation of Ta carbide on the surface of the PhC at high temperatures.<sup>13,18,19</sup> In this study an additional coating of 40 nm  $\text{HfO}_2$  is deposited on the fabricated PhCs by atomic layer deposition (ALD, Cambridge NanoTech Savannah) at a substrate temperature of  $250^\circ\text{C}$  using (Tetrakis)dimethylmino Hafnium and water as a precursor. The ALD deposition ensures completely conformal deposition of the coating in the high aspect ratio cavities.

### 3. RESULTS

#### 3.1 Photonic Crystal Characterization

The Ta 3% W PhC is designed for a cutoff wavelength  $\lambda_{cutoff} = 2.0\ \mu\text{m}$  which corresponds to a band gap of 0.62 eV (InGaAs TPV cell). The optimized geometrical parameters for this case are a period  $a = 1.3\ \mu\text{m}$ , radius  $r = 0.54\ \mu\text{m}$  and cavity depth  $d = 8\ \mu\text{m}$ . In the optimization, fabrication constraints were taken into account by ensuring a minimum space between the cavities and limiting the cavity depth. The benefit of increased selectivity of the emittance diminishes for cavity depths above  $d \sim 8.0\ \mu\text{m}$ . The measured cavity radius of the fabricated PhCs was  $r = 0.53\ \mu\text{m}$ , with a period  $a = 1.37\ \mu\text{m}$ . The depth is experimentally measured by milling into the substrate using a focused ion beam (FIB) microscope. In order to achieve a cleaner cross-section, the cavities are filled with Pt and then milled. The etch depth measured by FIB was  $6\ \mu\text{m}$  on average, corresponding to an aspect ratio of 6 to 1. In our previous study a grass formation was present inside the cavity.<sup>20</sup> This effect was since reduced by optimizing the etch parameters, as shown in Fig. 3 where a PhC with  $r = 0.48\ \mu\text{m}$  and  $a = 1.0\ \mu\text{m}$  was fabricated with the same method as described in Section 2.2.

The simulation using the previously measured cavity radius and period of the fabricated PhC ( $r = 0.53\ \mu\text{m}$  and  $a = 1.37\ \mu\text{m}$ ) and a cavity depth of  $d = 6.7\ \mu\text{m}$  shows good agreement to the emittance obtained from room

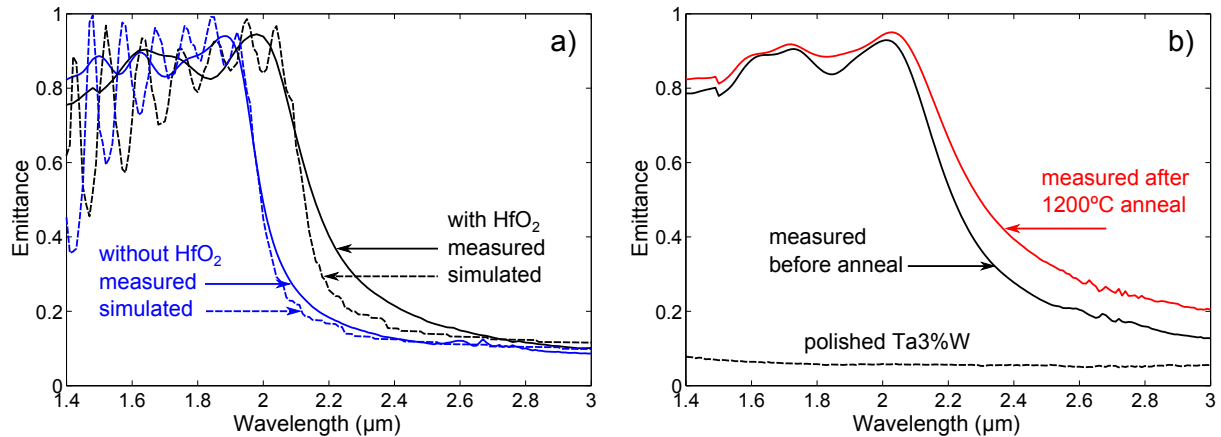


Figure 4. (Color online) (a) Comparison of the emittance obtained from reflectance measurements at room temperature of the Ta 3% W PhC without and with an  $\text{HfO}_2$  coating of 40 nm thickness, and simulations using a period  $a = 1.37 \mu\text{m}$ , cavity depth  $d = 6.7 \mu\text{m}$ , and radius  $r = 0.53 \mu\text{m}$  and radius  $r = 0.57 \mu\text{m}$  respectively for the uncoated and coated PhC. (b) Comparison of the emittance of the Ta 3% W PhC with an  $\text{HfO}_2$  coating before and after anneal at  $1200^\circ\text{C}$  for 24 h in a vacuum furnace with the emittance of the polished substrate.

temperature reflectance measurements as shown in Fig. 4 a) with a cutoff wavelength of about  $\lambda_{cutoff} = 2.0 \mu\text{m}$ , demonstrating high emittance below the cutoff wavelength while maintaining low emittance above, with a steep cutoff between. The spectral efficiency (defined as the ratio of useful emission to total emission) can be found from the simulated hemispherical data using high temperature material properties. Very good selectivity is observed for the PhC emitters which increases with temperature as expected. The total calculated hemispherical spectral efficiency is found to be greater than 50% above  $\sim 1100^\circ\text{C}$ . System modeling shows that the expected TPV thermal-to electrical efficiency using an InGaAsSb TPV cell and a matched PhC emitter (with no further spectral components such as filters) can reach 29.1% for ideal TPV cell properties, and 13.0% for actual measured TPV cell properties, both at an operating temperature of  $\sim 1170^\circ\text{C}$ .<sup>21</sup>

### 3.2 Emittance with and without $\text{HfO}_2$ ALD layer

As mentioned in Section 2.2, surface diffusion and surface chemical reactions are known high-temperature PhC degradation mechanisms and previous studies have shown a protective dielectric coating such as  $\text{HfO}_2$  can reduce these unwanted effects. In this study a protective coating of 40 nm  $\text{HfO}_2$  is used in an attempt to prevent structural degradation by surface diffusion, grain boundary grooving, as well as surface reactions such as the formation of Ta carbide on the surface of the PhC at high temperatures. While the additional layer influences the emittance, causing the cutoff wavelength to shift slightly to a higher wavelength, the sharp cutoff, low emittance at longer wavelengths and high selectivity are preserved. Fig. 4 a) shows the emittance of the fabricated PhC with and without the coating as obtained from room temperature (RT) reflectance measurements, showing a shift of the cutoff from 2.0 to 2.15  $\mu\text{m}$ . This shift of the cutoff wavelength is reproduced by simulations of the PhC with the coating. For simplification, it can be taken into account in the simulations by using a larger cavity radius  $r$  as the optical path length in the cavity is effectively increased by the addition of a high index material ( $n \sim 1.8$ ). Thus, to achieve the target cutoff wavelength with the coated PhC a slightly smaller cavity radius has to be fabricated than given by the optimization without the coating.

To study the thermal stability, the fabricated PhCs were annealed at  $1200^\circ\text{C}$  for 24 h in a vacuum setup under Ar atmosphere ( $10^{-7}$  Torr base pressure,  $10^{-4}$  Torr flowing UHP Ar). To prevent convection and conduction losses, as well as oxidation and degradation of the microstructured materials, any high-temperature TPV system is preferably run under vacuum. The reflectance of the Ta 3% W PhCs with the  $\text{HfO}_2$  ALD coating was obtained experimentally at room temperature before and after annealing by spectroradiometric measurement. Note that the emittance can be obtained from the measured reflectance by using Kirchhoff's law ( $E = 1 - R$  for non transmitting substrates). As shown in Fig. 4 b), the spectral emittance of the Ta 3% W PhC and its selectivity

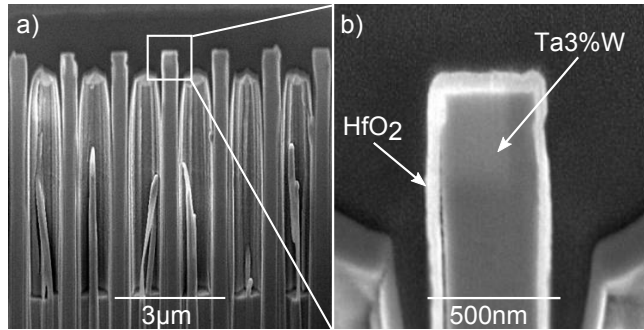


Figure 5. FIB cross section of the Ta 3% W PhC after 24h anneal at 1200°C for 24 h in a vacuum furnace. Note that Pt was deposited prior to FIB milling in order to improve the cross-sectional imaging. The inset is a close up of the cavity wall.

is essentially preserved after the 24 h anneal at 1200°C. A small degradation of the cutoff tail is observed after anneal, as well as a slight increase of the emittance above the cutoff wavelength. The furnace used was limited to 1200°C preventing us to go to higher temperatures, but longer term annealing studies are under way.

Thermal degradation of the reflectance spectra could be due to a rounding of structure profiles due to surface diffusion or surface reactions like carbide formation, as observed in a previous study on Ta samples without coating.<sup>13</sup> The structural stability of the Ta 3% W PhC with the HfO<sub>2</sub> ALD coating was studied through the FIB cross sectional images before and after annealing. Very little change was observed after annealing at 1200°C for 24 h under Ar atmosphere, as shown in Fig. 5. Therefore we conclude that surface diffusion was effectively prevented by the HfO<sub>2</sub> coating at least for the observed time and temperature scale. We could, however, observe the beginning of localized carbide formation on the surface of the HfO<sub>2</sub> coating, as confirmed by Auger spectroscopy. This is the reason for the degradation of the emittance spectrum of the PhC and thus a surface passivation carbide barrier is needed. Although the reaction is significantly slower than on the Ta surface (without the coating) the carbide formation at higher temperatures could eventually compromise the spectral selectivity of the emitter. Further investigation on the source of carbon and the prevention of this reaction is under way.

#### 4. CONCLUSION

In conclusion, in this paper a tantalum tungsten solid solution alloy, Ta 3% W, was investigated as a substrate for 2D PhCs for high-temperature energy applications. The Ta 3% W alloy presented advantages compared to the non-alloys as it combined better high-temperature thermo-mechanical properties of W with the more compliant material properties of Ta, allowing for a direct system integration path. The mechanical stability of the selective emitters as part of a high temperature system was found to be greatly enhanced using the Ta-W alloy substrates instead of pure Ta, reducing potential degradation such as creep and deflection which can play a critical role in system failure. Furthermore, Ta 3% W is advantageous as the thermo-mechanical properties can be tuned by the W content. A 2D PhC on a Ta 3% W alloy substrate was designed using FDTD numerical simulations and its spectral emittance was optimized to match a PV cell with a bandgap at 2.3 µm. The PhC emitter was fabricated using a process similar to that used previously for Ta PhCs, however the fabrication process was further tuned to minimize grass effects and results in PhCs with unprecedented uniformity. The emittance of Ta 3% W was obtained from near-normal reflectance measurements at room temperature before and after annealing at 1200°C for 24 h in vacuum with a protective dielectric coating of 40 nm HfO<sub>2</sub>, showing high spectral selectivity as predicted by simulations. A slight degradation of the emittance spectrum was attributed to the beginning of carbide formation on the surface of the HfO<sub>2</sub> coating, which is greatly decelerated in contrast to the surface reaction on Ta without the coating. At this time, it still appears that the observed surface degradation is only due to contamination, but more tests are being conducted in regards to substrate preparation. Scanning electron microscope images of the cross section of the PhC prepared by focused ion beam milling compared before and after anneal confirm the structural stability of the HfO<sub>2</sub> coated Ta 3% W PhC and no degradation, i.e. rounding

of the profile, was observed, which confirms that the coating effectively prevents structural degradation due to surface diffusion. All in all, these results confirm that Ta 3% W based PhCs with an HfO<sub>2</sub> coating are a promising solution for spectrally selective optical components for high-temperature applications.

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