High-temperature resistive surface grating for spectral control of thermal radiation

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(Received 26 August 2002; accepted 21 January 2003)

Spectral emittance and thermal stability of two-dimensional W gratings are investigated to obtain high-temperature resistive selective emitters. Numerical calculations based on rigorous coupled-wave analysis are performed to determine the structural profile of gratings with good spectral selectivity. According to the determined parameters, two-dimensional W gratings composed of rectangular microcavities with the period of 1.0 μm are fabricated on single crystalline and polycrystalline W substrates. The grating shows a strong emission peak which can be explained by the confined modes inside the cavities. The grating with 200 nm wall thickness made from a single crystalline W shows very high thermal stability over 1400 K, while the polycrystalline grating is deformed at a high temperature because of the grain growth. [DOI: 10.1063/1.1560867]

Recently, spectral control of thermal radiation has been focused on as an application of micromachining techniques. This method is expected to be applied in various thermal systems, since it has potential advantages such as adjustability of spectral design and raw material. Up to now, several groups have demonstrated spectral control of emittance by means of periodic microstructures: one-dimensional SiC gratings,[1,2] two-dimensional (2D) metallic gratings,[3–6] and three-dimensional photonic crystals.[7,8] On the other hand, thermal stability of such microstructures, which is a critical problem in case this method is applied in a high-temperature environment where thermal radiation mostly dominates heat transfer phenomena, has not been discussed sufficiently yet. In this letter, we report that superior thermal stability of surface microstructures can be achieved by using single crystalline metals. 2D surface gratings with 200 nm wall thickness on single and polycrystalline W show strong emission peaks due to the standing wave resonance inside cavities. The single crystalline grating keeps its spectral feature as well as microstructure after a heating test up to 1400 K, while deformation of the microstructure is observed on the polycrystalline grating.

In this study, we take up selective emitters for thermophotovoltaic (TPV) generations,[9] which are operated at 1000–1500 K, as an example in various applications of spectrally selective devices used at high temperatures. In TPV systems, it is necessary that the spectral property of emitters is matched with the spectral response of photovoltaic cells. For instance, the sensitive region of GaSb cells is about 0.8–1.8 μm, and this requires emitters to have a high emittance only within this band and low outside it. To design a selective emitter for GaSb cells, we have performed numerical calculations based on rigorous coupled-wave analysis (RCWA).[10] We have chosen W as a raw material of gratings expecting a good thermal resistibility. Using its optical constants at room temperature reported in the literature,[11] we have simulated spectral absorptance of W gratings and regard it equal to their emittance according to Kirchhoff's law. Temperature dependence of optical constants is ignored in all calculations. Figure 1 shows calculated spectral emittance of W gratings composed of rectangular cavities as drawn in the inset. The cavities are placed periodically and symmetrically to the x and y directions. Figure 1(a) shows normal emittance spectra plotted for a few aperture size a, on the condition that period Λ and the aspect ratio a/d are fixed at 1.0 μm and 1.0, respectively. Spectral emittance of the gratings increases significantly with increasing a mainly in the wavelengths of about $0.3<\lambda<2.0$ μm. High emittance region broadens simultaneously. This behavior corresponds to lengthening the cutoff wavelength of the cavities, which are basically represented by $2a$. The result reveals that the emittance enhancement is related to the resonance between electromagnetic waves and standing wave modes inside the cavities.[5,6] In Fig. 1(b), calculated emittance spectra are plotted as a function of the incident angle θ with the fixed parameters, $\Lambda=1.0$ μm, $a/\Lambda=0.8$, and $d/a=1.0$. The spectral profile of the cavity array does not depend very much on θ, unlike to surface plasmon polaritons. From these results, we consider that the very high emittance in $\lambda<2.0$ μm is originated from the superposition of standing wave resonances inside the cavities and W's intrinsic absorption. Good spectral selectivity and angle-insensitivity are important in TPV selective radiators, and therefore we have chosen $\Lambda=1.0$ μm, $a/\Lambda=0.8$, and $d/a=1.0$ as parameters to fabricate samples.

2D microcavity arrays have been processed on W substrates by means of electron beam (EB) lithography and fast atom beam (FAB) etching.[12] First, a thick resist layer (thickness$=1.5$ μm), an Al thin film (100 nm), and an EB resist layer (0.5 μm) are stacked one after another on a mechanically polished W substrate. The top EB resist is exposed by an EB lithography system (ELIONICS, ELS-3700) and a grating pattern is drawn. After the development of the pattern, FAB etching with SF_6 gas is carried out with a FAB system (EBARA, FAB-60ML) to replicate it on the Al film.

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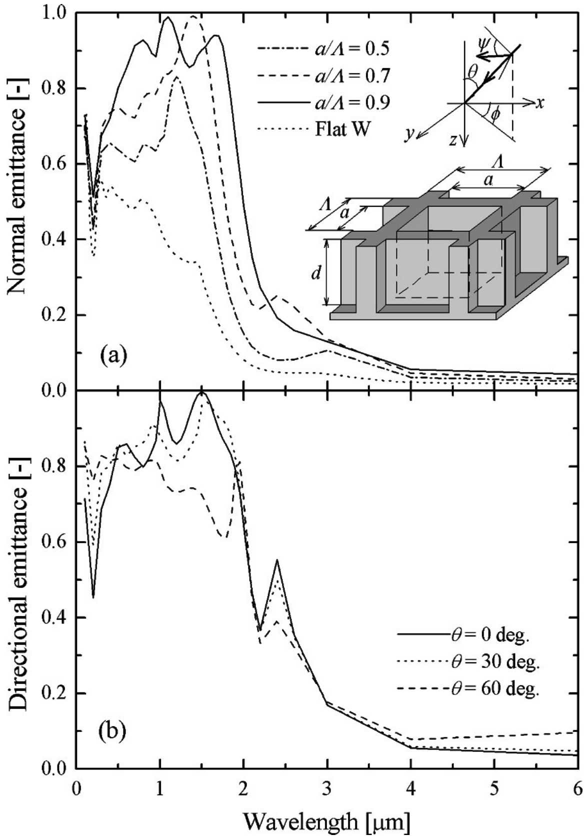

FIG. 1. Calculated emittance spectra of the 2D W gratings with rectangular cavities. Calculation model is shown in the inset including following parameters: period Λ, aperture size a, depth d, incident angle θ, azimuthal angle ϕ, and polarization angle ψ. In all calculations, $\Lambda = 1.0\ \mu$m, $d/a = 1.0$, $\phi = 0°$, and $\psi = 45°$. (a) Normal emittance of W gratings with the different value of a. (b) Incident angle dependence of spectral emittance of W grating with $a/\Lambda = 0.8$ and $d/a = 1.0$.

Since FAB is electrically neutral atomic or molecular beam, it is possible to obtain fine patterns with nanometer order without deformation of etching shape due to accumulated charge on samples. Next, FAB etching with O_2 gas is successively performed to replicate the pattern on the thick resist under the Al film. With this double layer method, we can obtain a deep resist mask. Finally, FAB etching with SF_6 is performed again and the grating pattern is transferred on the W substrate consuming the deep resist mask. Figures 2(a) and 2(b) show scanning electron microscope (SEM) images of 2D W gratings with $\Lambda = 1.0\ \mu$m processed on a single crystalline W substrate. a and d are determined as 0.8 and 0.75 μm, respectively, through SEM observations. It is observed that the cross section of each cavity is not a perfect rectangular but a trapezoid whose base length of the bottom is a little bit shorter than that of the top. In addition, the walls of each cavity are uneven, but the roughness is sufficiently smaller than Λ. We have also applied this procedure to a polycrystalline W substrate and obtained a grating whose surface structure is almost the same as shown in Fig. 2.

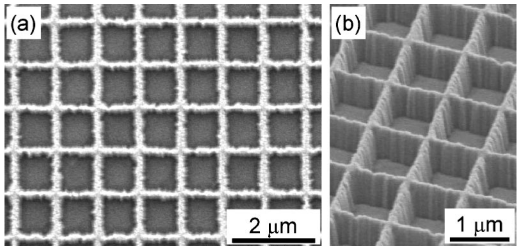

FIG. 2. SEM images of the 2D single crystalline W grating with $\Lambda = 1.0\ \mu$m. (a) Top view. (b) Oblique view.

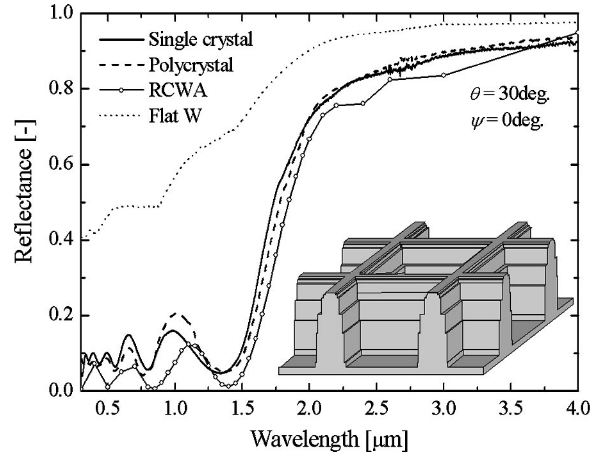

FIG. 3. Reflectance spectra of the W gratings measured at $\phi = 30°$ and $\psi = 0°$ with random polarized incident beam. Solid line: single crystalline grating, dashed line: polycrystalline grating, dotted line: flat W. Solid line with circles represents spectral reflectance calculated with the more realistic calculation model as drawn in the inset.

Before measuring thermal emittance, we have measured spectral reflectance of the single and poly crystalline W gratings. The results are plotted in Fig. 3. Irrespective of crystallinity, reflectance of each grating decreases drastically at $\lambda < 2.0\ \mu$m keeping high reflectance at longer wavelengths. Compared with Fig. 1, spectral selectivity of the samples is somewhat worse than the calculated spectra. This can be explained by the difference of the cavity shape. Actually, we have carried out RCWA calculations with a realistic model as shown in the inset and obtained the reflectance spectrum which agrees well with the measured spectra. A clear minimum is observed at about 1.35 μm in the measured and calculated spectra. Calculations reveal that that wavelength does not depend on θ very much like Fig. 1(b). So this minimum is probably due to the standing wave resonance inside the cavities.

We have measured thermal emittance spectra of the

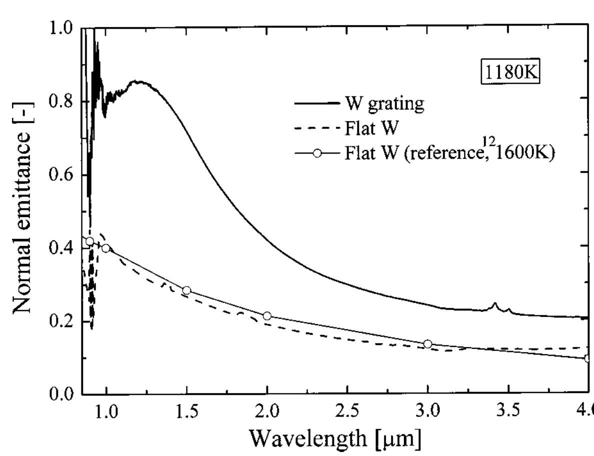

FIG. 4. Measured normal emittance spectra of the single crystalline W grating. A significant peak appears at about 1.25 μm. Solid line with circles represents a reported emittance spectrum of flat W.

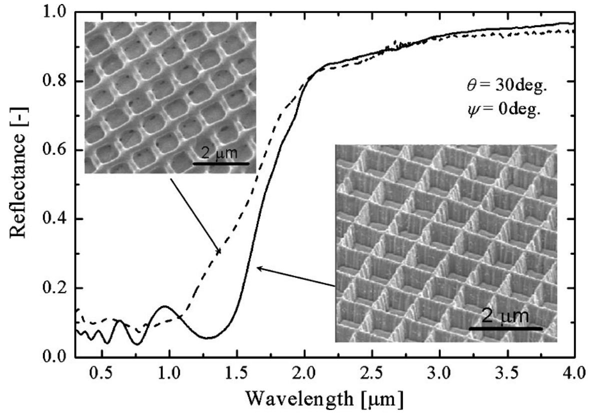

FIG. 5. Reflectance spectra of the W gratings after a heating at 1400 K for 1 h in a reductive atmosphere. The single crystalline grating keeps its spectral feature (solid line) and microstructure as shown in the inset. But the spectral property of the polycrystalline sample becomes worse than that of before heating (dashed line). Its microstructure is also damaged as shown in the inset.

samples with the emission measurement system equipped with an electric heater, a Fourier-transform near-infrared spectrometer with an emission port (Perkin Elmer, GX2000), and some optical elements. The measured spectra are calibrated and given units by comparing with the emissive spectra of a material whose emittance has been already known, for example, SiC. During heating samples, $5\%H_2-Ar$ gas is flown inside the heater casing to prevent samples from oxidation. Surface temperature of microstructured samples is determined with a radiation thermometer by measuring the temperature of a flat W heated simultaneously with them. Figure 4 shows the normal emittance spectra of the single crystalline W grating shown in Fig. 2 and a flat W substrate at 1180 K. Literature data[13] of spectral emittance of flat W are added in the figure to check the accuracy of measurements. Emittance of the grating increases drastically in the shorter wavelengths as expected from the calculation results. It is considered that a peak appeared at about 1.25 μm corresponds to the reflectance minimum in Fig. 3, but the wavelength is slightly shorter than that of the reflectance minimum. In addition, the sum of emittance and reflectance is not unity at some wavelengths. These probably derive from temperature dependence of optical constants, which is ignored in our calculations. The samples have been heated up to 1400 K. The spectral feature of the single crystalline grating has hardly changed at 1400 K, while that of the polycrystalline grating has been aggravated with increasing temperature.

Figure 5 shows the reflectance spectra of the gratings after a heating at 1400 K for an hour in $5\%H_2-Ar$ atmosphere. SEM images of them are inset in the figure. The single crystalline sample keeps almost perfectly its original microstructure composed of 200 nm thickness walls and spectral properties. On the other hand, the polycrystalline grating displays degradation of spectral selectivity, which is caused by a severe deformation and creation of voids as shown in the inset. Significant grain growth is also observed on the polycrystalline sample. Considering these results and the very high melting point of W, we regard the deformation of the polycrystalline W grating is mainly caused by the fast atomic diffusion at grain boundaries. From these results, it is confirmed that single crystalline metal is very useful for fabricating high-temperature resistive microstructures.

In our summary, we have shown concretely the effect of crystallinity to the thermal resistibility of 2D surface gratings with 1.0 μm periodicity composed of microcavities fabricated on both of single and polycrystalline W substrates. The thermal resistivity of the spectral feature as well as microstructures has been drastically improved by using single crystalline substrates. The thermal stability up to 1400 K is enough to be applied to selective emitters for TPV generations. This knowledge will be a breakthrough for the future developments of micro- and nanotechnology, especially in high-temperature energy conversion fields.

The authors appreciate the monetary support by the Ministry of Education, Science, Sports and Culture, Japan, Japan Space Forum, and the Asahi Glass Foundation. The authors are also grateful for the technical support by the Venture Business Laboratory in Tohoku University.

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