



Direct current reactive sputtering Cr–Cr₂O₃ cermet solar selective surfaces for solar hot water applications

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ARTICLE INFO

Article history:

Received 30 July 2008

Received in revised form 16 September 2008

Accepted 18 September 2008

Available online 30 September 2008

Keywords:

Solar thermal selective surfaces

Black chrome

Reactive sputtering

Solar hot water collectors

ABSTRACT

Replacing conventional electroplating method in manufacture of black chrome solar selective surfaces with reactive sputtering technique is desirable due to the environmental concern in order to meet rapid expansion of utilization of solar hot water systems. Experimental investigation revealed that the rapid transition region in reactive sputtering from a pure chromium metal to oxygen-rich materials resulted in the difficulty of optimization. We proposed a low-high-low (or with further repetition if needed) metal volume fraction profile for the cermet layer and a method using the target current as a control parameter in optimization. The solar absorptance of the sputtered black chrome selective surfaces was in the range 0.92–0.96 and the thermal emittance was between 0.05 and 0.08. Accelerated aging test showed that the surfaces were thermally stable to approximately 300–400 °C. Aging continuously at 170 °C in ambient environment for a period equivalent to 17 years in extreme stagnation condition, the surfaces did not show noticeable degradation. The developed method offers additional freedoms for designing new solar selective surfaces using non-conventional materials.

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1. Introduction

Black chrome, a form of nano-sized chromium particles mixed with chromium oxides, has been utilized for more than 20 years as solar hot water absorbers using electroplating method [1–3]. Nowadays, a significant proportion of flat solar hot water panels are manufactured using this method. As the solar hot water systems have the highest energy conversion efficiency in known solar energy device technologies because of the direct energy conversion at relatively low operation temperatures, the use of more and more solar hot water panels becomes a clear trend for establishing an energy sustainable society and slowing down global warming effect. It therefore results in demanding of increasing the capacity of electroplating manufacture for solar hot water absorbers. However, electroplating has caused widespread environmental concerns. In the broadest sense, the electroplating technology for sustainable energy is not sustainable. In deed, more and more stringent environmental regulations have been implemented, which has restricted the electroplating industry to expand the manufacture scope worldwide. As the black chrome solar selective surface is well accepted as solar hot water absorbers, it will be greatly useful to have an alternative method for deposition of black chrome type surfaces.

Direct current (DC) sputtering has been used in reactive sputtering mode to produce solar cermet surfaces using other metals [4–6]. In literature, though Cr–Cr₂O₃ cermet selective surfaces can be well suited for solar thermal applications, relatively fewer work was reported on black chrome or Cr–Cr₂O₃ cermet solar selective surfaces using DC sputtering technique. Nunes et al reported [7,8] that they obtained Cr–Cr₂O₃ cermet selective surfaces with absorption 0.94 and emittance 0.06 without giving detained result to show the solar selectivity in wavelength range from visible to far infrared. Recently, Barshilia et al, attempted solar selective surfaces with multilayer of chromium and chromium oxide using a pulsed sputtering method with solar absorptance 89–91% [9]. Pulsed sputtering technique compared with DC sputtering method, has considerably lower deposition rate thus it is disadvantaged for large scale and low cost solar hot water absorber manufacture. In addition, multilayer of chromium and chromium oxide has to consist of a relatively very thin chromium layer (typically about a few nanometres), which results in more restricted process control and more vulnerable to aging. Lee reported [10] DC sputtering deposition of chromium oxide cermet solar selective surfaces based on two chromium oxide cermet layers on aluminium reflecting metal layer. The introduction of aluminium instead of chromium as a reflecting layer makes this method questionable for replacing conventional black chrome surfaces because aluminium reflector layer cannot be as stable as chromium in ambient condition. It is known that aluminium surfaces will be oxidized gradually in ambient condition so that degradation of the solar selective surfaces will occur. The introduction of the aluminium

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reflecting layer could result in complication of process as the emissivity of the selective surfaces reported by Lee [10] was about 0.10, much higher than the emissivity of the base reflecting layer, indicating the degree of difficulty encountered during optimization. Though solar selective surfaces with aluminium reflecting layer can be used in vacuum glass tubular collectors, the high emissivity reduces solar thermal efficiency considerably compared with conventional selective surfaces for vacuum glass tubular collectors. It is therefore necessary to study further DC reactive sputtering solar Cr–Cr₂O₃ cermet selective surfaces based on chromium reflecting layer for direct comparison with electroplating black chrome selective surfaces.

In solar hot water absorber designs, metal volume fraction profile in cermet layer is one of the main parameters to consider in design. The volume fraction of metal to dielectric material can be adjusted from the top surface to substrate to optimize for solar spectrum selectivity and solar thermal efficiency, i.e. to achieve the maximum absorbance in solar radiation spectrum region and maximum reflectance in the thermal radiation region (typically at wavelengths beyond 2 μm). The proposed metal volume fraction profiles include mainly graded and multilayer profiles. In the graded profile, the cermet metal volume fraction increases gradually for the top surface to substrate. In multilayer profile, the cermet metal volume fraction increases from the top surface to substrate with two or more steps. The approach monotonously increasing metal volume fraction from the top surface to substrate could have placed a non-necessary restriction on solar selective surface design. In this work, we proposed a method to allow the cermet metal volume fraction to decrease and also to increase from the top surface to substrate.

A related method was previously proposed [11], in which the metal volume fraction profile [11] was a high metal volume fraction layer placed onto the top of a low metal volume fraction layer (HL structure). In their report, the reported theoretically simulated profile was a 10 nm thick layer with 0.25 metal volume fraction placed onto a 70 nm thick layer with 0.20 metal volume fraction, using copper as the metal and silicon oxide as the dielectric material. They [11] also showed one experimental example of such selective surfaces, claimed that both the thickness and volume fraction were very close to their simulated value. The reported performance of the solar selective surface with the HL structure was obviously lower than that of their typical surfaces with LH structure, for both simulated and experimental selective surfaces. It is an interesting question whether the difference between the high volume fraction (0.25) and low volume fraction (0.20) is significant, and how sensitive to the change of the thin layer thickness (10 nm) of the high volume fraction layer. However, no further work was reported for such comparisons.

The aim of this work is to use the DC sputtering method for deposition of Cr–Cr₂O₃ cermet (or black chrome type) solar selective surfaces by combining the proposed cermet metal volume profile that allows metal volume fraction to decrease as well as to increase from top to substrate in the cermet layer range. The refractive index of the cermet materials is also to be characterized for understanding of the Cr–Cr₂O₃ cermet formation mechanism in the reactive sputtering system. The performance of the deposited Cr–Cr₂O₃ cermet (or black chrome type) solar selective surfaces is also to be investigated to provide comparison to conventional.

2. Experimental details

The dual-cathode DC sputtering system used in this work is schematically shown in Fig. 1. The DC sputtering system was manufactured by JAVAC Pty Ltd, Melbourne, Australia. The deposition chamber was a cylinder with height of 600 mm and diameter of 650 mm. The substrates were placed at the distance of 12 cm from sputtering the target. The purity of argon was 99.999%. The reactive gas for deposition of Cr–Cr₂O₃ cermet was 10% oxygen mixed with 90% argon with purity of 99.99%. The purity of the chromium target was

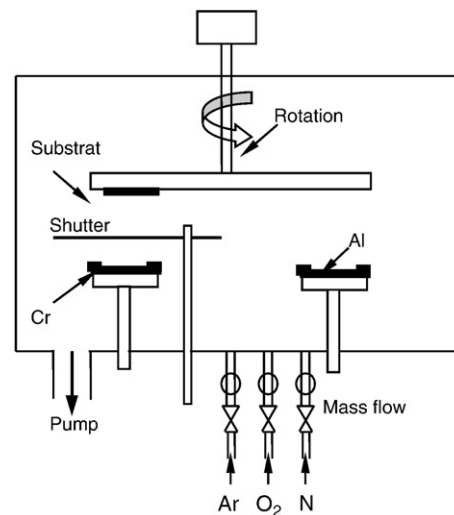


Fig. 1. Schematic diagram of the DC sputtering deposition system.

99.9% from Goodfellow Corporation, UK. The purity of the aluminium target was 99.8%. The aluminium target was used for deposition of an anti-reflection layer on cermet surfaces. The base pressure of the system was 4×10^{-4} Pa. During the sputtering the pressure was maintained at 3×10^{-1} Pa. Optical slides were used as substrate. The refractive index of deposited thin films was characterized using a spectroscopic ellipsometer (J.A. Woollam M-2000) in the wavelength region between 400 and 1000 nm. Spectrometer Cary-5E and Shimadzu IR-470 were used to characterize the reflectance in the wavelength region from 300 nm to 25 μm. Solar absorbance was calculated using standard AM1.5 solar spectrum, and thermal emittance was calculated using the experimental reflectance data and blackbody radiation spectrum at 100 °C. The reflecting layer prior to deposition of cermet layer was a pure chromium thin film with thickness between 200 and 400 nm.

In this work, the dependence of refractive index of the cermet materials was characterized as a function of basic deposition parameters: oxygen flow rate and sputtering cathode current. The solar thermal stability of the solar selective surfaces was characterized in two conditions: the accelerated aging by annealing at temperature between 200 and 500 °C for 1 h; and the long time aging (over two years) at 170 °C in interior ambient environment without humidity control.

3. Results and discussion

Fig. 2 shows the dependence of chromium cathode voltage as a function of oxygen flow rate. The chromium target current in this case was 0.2 A. The total gas flow rate was maintained at 13 standard cubic centimeter (sccm). In the range between 0.3 and 0.6 sccm, the cathode voltage changed rapidly and peaked near 0.5 sccm. The dependence of the cathode voltage to reactive gas flow rate was generally discussed by Hohnke et al. [12]. The rapid change of the voltage is correspondent to the so-called cathode “poisoning effect”, in which the reaction rate of the reactive gas starts to exceed the sputtering rate of the compound on the target. On the left side of the transition region, it produces materials with rich metal content, and on the right side it produces oxygen-rich or pure dielectric materials. The rapid transition behavior indicates that in this range the cermet metal volume fraction of this material would be difficult to control.

The “poisoning” effect of the Cr target can be also found in the dependence of deposition rate on oxygen flow rate as shown in Fig. 3. Similar to the dependence of cathode voltage on oxygen flow rate,

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