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An application of Au thin-film emissivity barrier on Ni alloy

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ABSTRACT

1000 nm-thick Au film was sputter-deposited on two groups of nickel alloy substrates, in which one group (Group A) was oxidated at 800 °C for 20 h to form a oxide film before coating gold while another group (Group B) was unoxidated. The gold thin-film is applied to serve as a low emissivity coating to reflect thermal radiation. The gold-coated samples were heated in air at 600 °C for 150 h to explore the effect of high-temperature environment on the emissivity of coated Au film. After heat-treatment, the average thermal emissivity at the wavelength of 3–14 μm of Group B greatly increased from 0.18 to 0.82 while that of Group A only increased a little. The diffusion between Au and other nickel alloy elements at 600 °C also had been discussed in this paper.

1. Introduction

Aerospace materials used in high temperature environments generally require thermal insulation and emissivity barriers to reduce the amount of absorbed thermal radiation [1]. Usually, the thermal emissivity of a clean metal surface is very low and stable, but it becomes to a high value when the surface is oxidized [2,3]. The thermal emissivity of metals is strongly affected by the growth of oxide films on the surface of metal [4]. Therefore, the non-oxidizable noble metals such as Au, Pt, Pd are the good choices for the emissivity barriers. Thin gold films have been used to provide low emissivity coatings on stainless steel, titanium and nickel alloys. In addition, aerospace service conditions are often harsh and materials may degrade after an extended service life. Some aerospace materials are regularly subjected to high temperatures, often with exposure to corrosive liquids and gases. Nickel alloys work well in these kinds of environments, but gold is much more resistant to corrosion from hydraulic fluids and fuels. High temperature components requiring corrosion resistance have an increased life span when gold is coated. The low emissivity value of the gold thin-film reduces the amount of thermal radiation absorbed and increases the materials in service flight time. The durability and strength of the nickel alloys are leveraged and the reduced labor costs are realized in the process.

Unfortunately, there is a serious problem for the use of gold film emissivity barrier on nickel alloy. The metal atoms in the nickel alloy can easily diffuse into the Au film at high temperatures [5]. If the diffusion time is long enough, the metal atoms will diffuse to the surface and then be oxidized. This situation is a catastrophe towards the application of emissivity barrier because of the very high thermal emissivity value of the metal oxide. The diffusion between Au and other metals at low temperature for 0.1–2 h has been widely studied [6–10], but there are few reports for the diffusion of Au at high temperature for a long time. In this paper, the properties of Au thin-film emissivity barrier using at high temperature were studied and the diffusion between Au and other metal atoms in nickel alloy at 600 °C for 150 h were also explored.

2. Experimental

The Ni-based alloy K424 is a high temperature alloy mostly consisting of Ni, Co, Cr, with trace amounts of Al, Ti, Mo and Fe. The samples with a size of about 15 mm × 15 mm × 1 mm are separated into two groups. One group (Group A) was oxidized at 800 °C for 20 h to form a oxide film before deposition of gold while another group (Group B) was deposited with gold directly

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without oxidization. Before deposition, both groups of substrates were ultrasonically rinsed in acetone, distilled water and ethanol, successively.

Au film with a thickness of about 1000 nm was deposited on the two groups by using an SBC-12-type DC sputtering system [KYKY] [11,12]. The deposition time of the Au layer was 50 min and the deposition rate was ~20 nm/min.

The two groups of gold-coated test panels were subjected to a constant high temperature exposure in air at 600 °C for 150 h, respectively to simulate the exposure of use environment.

The crystalline phase was examined by X-ray powder diffraction analysis using a Cu Kα radiation (Philips X'Pert diffractometer). The microstructure was observed with a scanning electron microscope (SEM) (model JSM-6360, Japan). IR emissivity values of the samples were determined using an infrared emissometer (SR5000 Israel).

3. Results and discussion

After the heat-treatment at 600 °C for 150 h, a distinct color change visible in the gold film was revealed. The color of Group B changed from gold to grey while the appearance of Group A was still golden. Fig. 1 shows small angle X-ray diffraction patterns of the gold-coated samples after heat-treatment. It is clear that the surface of Group B (Fig. 1(b)) consists of pure solid solution—Au0.7Cr0.3 phases and no secondary phases can be found. The result indicates that the Cr atom has diffused into the Au lattice and formed solid solution—Au0.7Cr0.3, causing the color change of the surface. In addition, the pattern of Group A (Fig. 1(a)) consists of pure Au phases and no solid solution phases can be detected. It implies that there is no Cr atom diffused into Au lattice. It can be concluded that the oxide film between Au and nickel alloy of Group A can countercheck the diffusion of the Cr atom, which is the reason why the color of the surface on Group A did not change after the heat-treatment.

Fig. 2 shows SEM micrographs obtained for the surface of gold-coated samples after heat-treatment. It can be seen that the surface of Group B is hackly on which there are lots of bulky particles with a size of about 10–20 μm. These particles are conterminal and random distributed. Fig. 3(b) shows Energy Dispersive X-ray Detector spectrum of the heated Group B sample within a square region approximately 50 μm × 50 μm in size. A significant amount of Ni, Al, Au, Cr, Ti, Co and O was detected. The existence of O atom means that the surface has been oxidized and it seems that the bulk particles on the surface of Group B shown in Fig. 2(b) are the metal oxides. Therefore, it is clear that the metal elements in K424 diffused through the gold over-layer and reacted with O atom in air to form metal oxide on the surface.

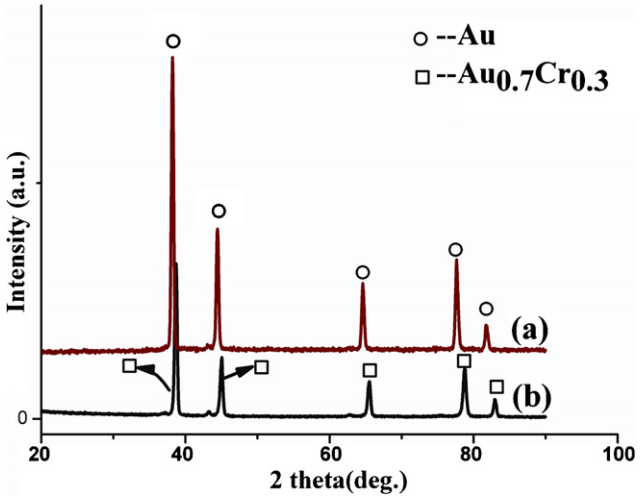

Fig. 1. XRD patterns of the gold-coated samples after heat-treatment at 600 °C for 150 h: (a) Group A and (b) Group B.

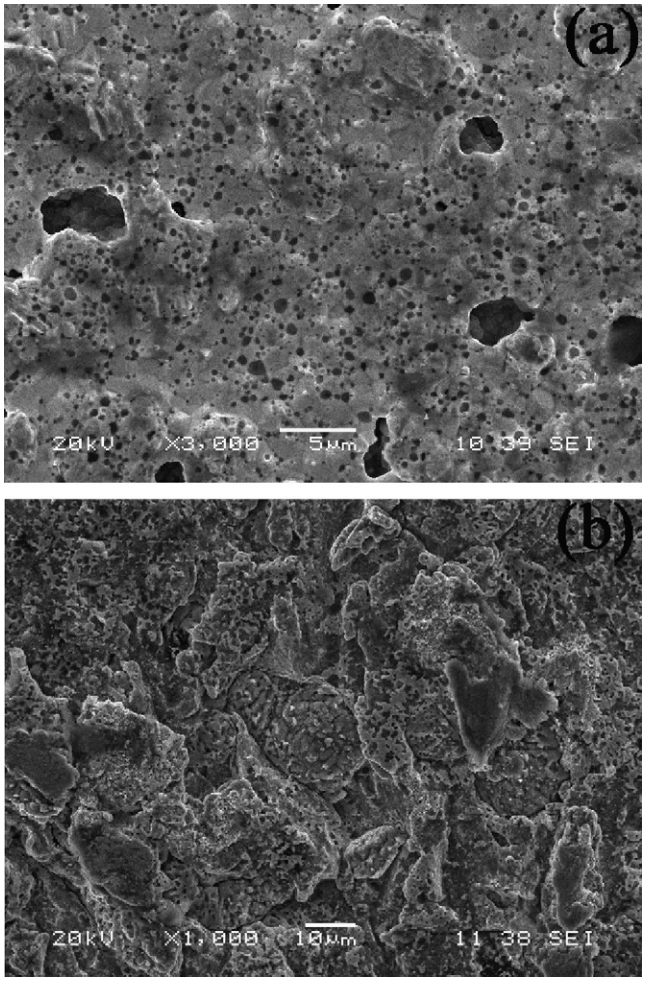

Fig. 2. SEM micrographs obtained for the surface of gold-coated samples after heat-treatment: (a) Group A and (b) Group B.

The SEM micrograph of the surface of Group A is shown in Fig. 2(a). It can be seen that the surface of Group A is smoother than that of the Group B. There are lots of pores on the surface of Group A, which were likely formed during the cooling. Fig. 3(a) shows Energy Dispersive X-ray Detector spectrum of the heated Group A sample. It can be seen that there is only the peak of Au, indicating that the surface is covered with almost pure Au. It is consistent with the above discussion about the XRD analysis. A trace amount of C and Ni is observed in Fig. 3(a). The C element is attributed to contamination that is commonly found on as received industrial metal surfaces [13]. The Ni atom inside the Au layer is about 6 at% and it probably originates from the nickel alloy.

Fig. 4(b) shows backscattered electron images of the cross-section for the Group B sample, in which the light areas correspond to Au whose atomic number is high. It is clear that the Au atom has interdiffused with the other substrate metal atoms, and it can be seen that the Au atom had deeply diffused into the nickel alloy through grain boundary. The Au layer on the surface of Group B disappeared after heat-treatment and the Au layer was replaced by the metal oxide film on the surface.