INVESTIGATION OF CHEMICAL VAPOR DEPOSITION PROCESSES TO PERFORM DENSE α-ALUMINA COATING ON SUPERALLOYS

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Coated nickel-based superalloys are widely used as superior material systems in gas turbines, where the overlay coatings must withstand hot corrosion and serve as bond coats for the thermal barrier layer. However, interdiffusion may limit the coatings' lifetime, a potential remedy being a diffusion barrier layer. In this context, the CVD process was investigated to deposit α-Al_2O_3 on superalloy substrates (CMSX-4).

Deposition was carried out in a horizontal hot-wall reactor, at 1080 °C using the $AlCl_3/CO_2/H_2$ gas mixture with flow rates of 0.034, 0.04, and 0.2 slm, respectively. Achieved films at low pressure (5 mbar) were composed of pure α-Al_2O_3; however they presented whisker growth with an average diameter of 65 nm. Under these conditions, the CMSX-4 substrates underwent a selective oxidation of the Ti present in the surface and the precipitation of Co from the matrix of the Co-Ni solid solution. The attained structure of the films experienced some alterations depending on the substrate position and the $AlCl_3$ flow rate; however, the pressure variation was noted to be the most significant parameter. In fact, the gradual increase of the pressure was noted to favor the deposition of dense films, until a totally closed structure was achieved at 100 mbar. Films deposited under these conditions presented a dense structure formed by compact crystals with an average size of 2 µm, as shown in Fig. 1. Under these conditions, the CMSX-4 substrates did not exhibit any noticeable alteration. Depositing alumina films with comparable properties at atmospheric pressure is economically of superior importance; however, the resulting films were composed of large and non-compact grains. To overcome this limitation, deposition of a thin α-Al_2O_3 layer by a sol-gel process was thought to minimize the substrate's effect and to guide the formation of a smooth α-Al_2O_3 layer at atmospheric pressure. In fact, this process led to the formation of smooth coatings, as shown in the Fig. 2, with a substantial decrease of the grain size.

In an effort to reduce the heavy maintenance imposed by the use of $AlCl_3$, the potential of aluminum tri-isopropoxide (ATI) precursor to produce dense α-Al_2O_3 coatings on CMSX-4 was investigated in a cold-wall CVD reactor at 1050°C using an ATI/O_2 gas mixture with flow rates of 0.1 and 0.7 slm, respectively. The chosen reactor geometry and flow rate of the inlet gas were proved to be successful in limiting the precursor depletion, which is the major limitation of this process. Films deposited at 100 mbar and a substrate temperature of 1050 °C present compact crystals forming a dense coating as shown in Fig. 3. The XRD analysis, Fig. 4a, of coatings obtained at 1050°C shows the co-deposition of γ-Al_2O_3 with α-Al_2O_3, and further increase of the temperature is suspected to eliminate the γ-Al_2O_3 phase. Another limitation consists of the substrate oxidation during the first stage of deposition. This effect was lowered when a high deposition rate was used (50 mbar).

In conclusion, using $AlCl_3$ precursor in the hot-wall reactor at 100 mbar, Fig. 4b, led to the pure α-Al_2O_3 with suitable microstructure without affecting the CMSX-4 substrates.

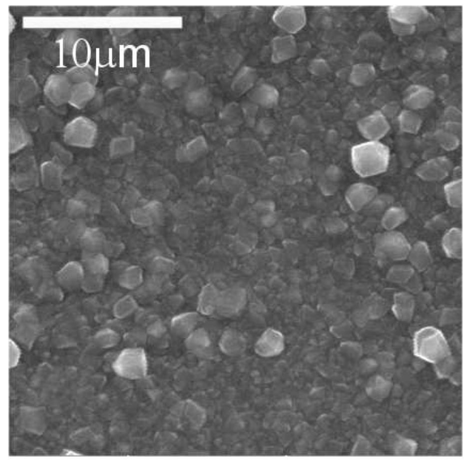

Fig. 1: SEM micrograph of α-Al_2O_3 film deposited on CMSX-4 (100 mbar, 2 h, 0.034 slm of $AlCl_3$).

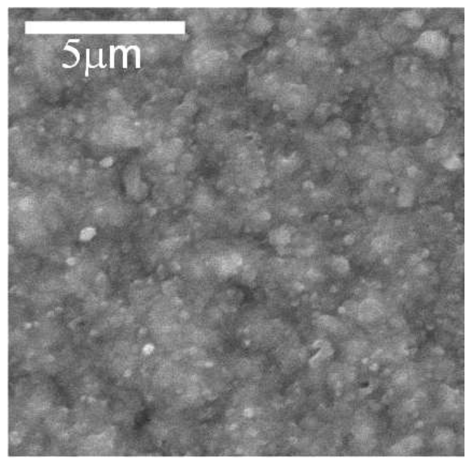

Fig. 2: SEM micrograph of α-Al_2O_3 film deposited on CMSX-4 using an intermediate alumina layer with a sol gel process (1 atm, 2 h, 0.034 slm of $AlCl_3$).

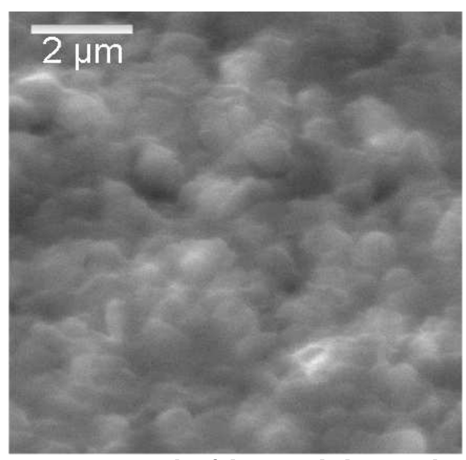

Fig. 3: SEM micrograph of deposited alumina thin film at 1050°C (100mbar, 0.1 slm of ATI).

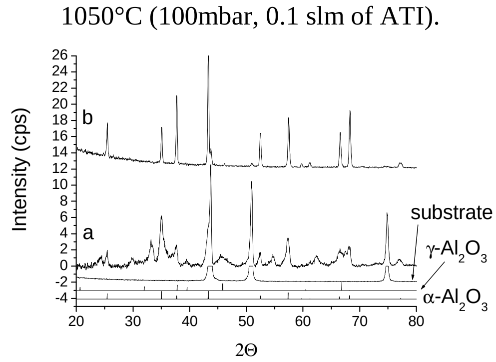

Fig. 4: XRD pattern of a film deposited at 1050 °C and 100 mbar in the cold-wall CVD reactor using ATI/O_2 gas mixture (a), and at 1080 °C and 100 mbar in the hot-wall CVD reactor using $AlCl_3/CO_2/H_2$ gas mixture.