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Thermionic vacuum arc and plasma spray processing of high temperature resistant coatings

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Abstract

A combined coating technology was developed using thermionic vacuum arc (TVA) method for deposition of Re and Re-Ni-Cr films [1] followed by plasma spray of Ni-5Mo-5Al or Al_2O_3 powders at high velocity and high temperature. The composite multilayered films were developed in order to protect turbine blade material (Nimonic 80 super-alloy) against high-temperature oxidation. The pure TVA metal plasma was monitored and diagnosed using optical emission spectroscopy, the prepared films being characterized before and after oxidation by scanning electron microscopy (SEM), electron dispersive spectrometry (EDS), X-ray diffraction (XRD), gravimetric thermal analysis (GTA) and differential thermal analysis (DTA).

I. Multilayer deposition by TVA

Evaporation of Re, Ni and Cr vas performed depositing the components simultaneously from different sources (as shown in Fig. 1) each being made of a pure element supposed to be combined with the others. Changing the evaporation conditions and the source -substrate distance for each component the desired reciprocal concentration can be obtained in the deposited layer. Some

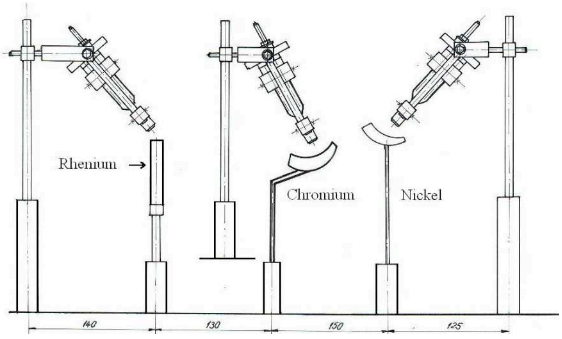


Fig.1 Schematic draw of the simultaneous deposition of Re-Ni-Cr

inconvenience appears because the deposition rates must be previously measured and connected with different physical parameters contributing to the evaporation of every element.

In order to produce plasma in pure Re vapours the thoriated tungsten filament of the TVA gun was heated by a 90-100 A current. The emitted electrons were focused on the Re anode by a Whenelt cylinder. The anode was made of a Re rod of 8-10 mm in diameter and supported by a Mo flange. Another W wire of 1.5 mm diameter and 100 mm length was used to sustain the previous ensemble and having the aim to avoid thermal energy flow. The distance between the thermoemissive filament and the Re anode was 3-4 mm and the angle between the electron beam and the vertical line was 60^0.

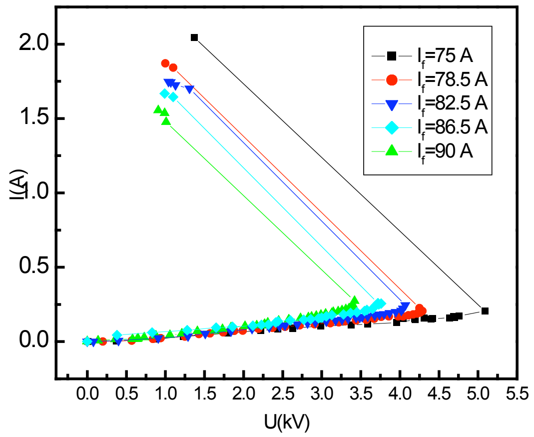


Fig.2. The V-A characteristics in Re vapours.

The anode applied voltage was increased up to 6000 V in that a way that when the upper part of the Re rod was heated up over 2500°C and the Re vapour pressure exceeded 1Pa, a bright electrical discharge was ignited. After the ignition, the discharge voltage decreased to the 1-1.5 kV as function of filament heating current. Fig. 2 shows the V-A characteristics, the voltages and currents of stable vacuum arcs in Re. In the same way the plasma was running in Cr, Ni or Ni-Al vapours. In this case the filament heating current was in the range of 17 - 26 A.

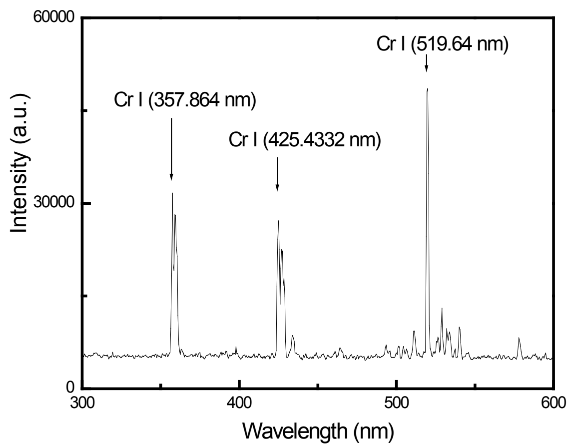


Fig.3 Optical emission spectrum of Cr plasma.

Using the ratio of the spectral lines intensities (the resonance emission lines Cr I 357.8684 nm and Cr I 425.4332) of the chromium plasma (emission spectrum shown in Fig.3) and assuming a partial local thermodinamic equilibrium (PLTE) [2], the electron temperature of the plasma running in pure chromium vapours was estimated to be in the range of 4240 – 4490 K.

The multilayer coating performed using TVA technology on the Nimonic 80 superalloy was: 200±50 nm Re, 500 ± 10 nm Re30Cr10Ni and 1000 ± 200 nm Ni20Al. Fig. 4 shows the surface morphology of the multilayer prepared by TVA and the EDS mappings of the deposited elements. (Re, Ni, Cr, Al)

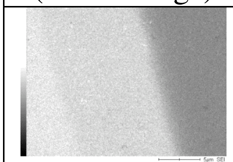
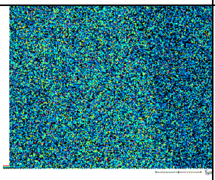
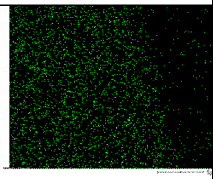
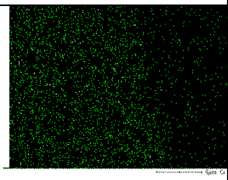
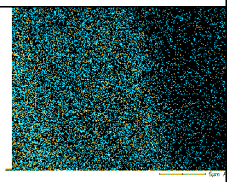
Surface morphology (SEM image)	Re (EDS map)	Ni (EDS map)	Cr (EDS map)	Al (EDS map)
				

Fig. 4. Surface morphology of the Re, Re30Cr10Ni and Ni20Al multilayer coating prepared by TVA (magnification: 1000 x)

Analyzing the XRD pattern of the Re5Cr, shown in Fig.5 can be assumed that the prepared film was policrystalline with large size Re crystals. The (002) peak of Re is higher than the theoretical one, meaning that a certain preferred orientation of crystals parallel with the substrate or/and the modification of the hexagonal Re network by the effect of a small quantity of Cr. The diffractogram of the Re30Cr (see Fig.6) shows a disordered structure, nearly amorphous, fact inferred by the broad peak at 21.5° (θ) and another very wide peak at 31° (θ).

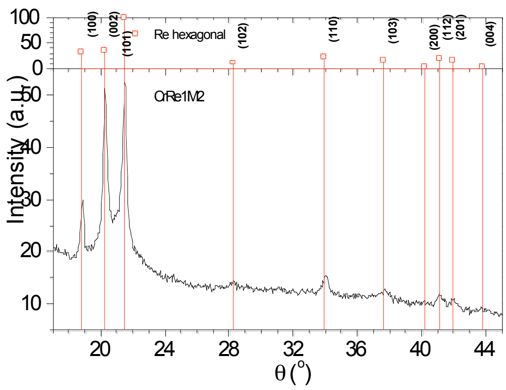


Fig.5. XRDpattern of the Re5Cr film

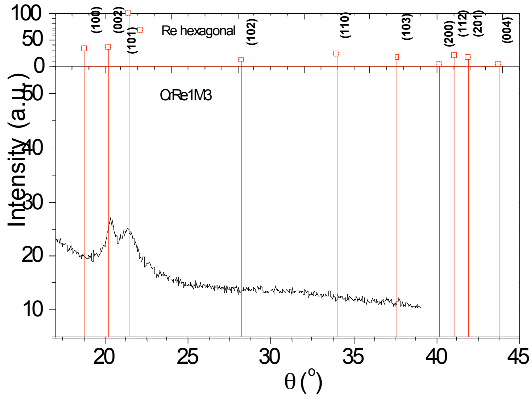


Fig.6. XRD pattern of the Re30Cr film

The lack of the Cr crystal phase can be explained by the amorphous phase formation, fact inferred by the raising of the XRD background in the 19.0-24.0 ° (θ) zone. In the same time with the raising if the Cr concentration the Re/Cr alloying took place and also the stabilization of the disordered amorphous phase.

The films were removed mechanically from the substrates and thermogravimetric measurements (TGA) and differential thermogravimetric analysis (DTA) were made using the following conditions: Temperature range: ambient ÷ 1273 K, Accuracy: 0,001 mg, Heating rate : 10 K/min; Maximum time at 1273 K:1 min; Working gas: air ; Reference material: Al_2O_3. The thermal stability of the ReCrNi film was higher in a sense that the endotherms and exotherm effects were drastically reduced with lower Cr concentration. The Re, Re-Ni-Cr and Ni-Al multilayer coating (1-2 μm), developed in the present investigation, containing around 70at% Re, was found to be more useful than the conventional Re-Ni-Cr film containing low percentage of Re. The Re and Re-Ni-Cr layers acts as a diffusion barrier between the Ni base super-alloy substrate and the 300 – 400 μm thick Ni-5Mo-5Al or Al_2O_3 layer, which forms a protective alpha-Al_2O_3 scale

II . Plasma spray coatings

In this work, a plasma spray evaporation method using a high velocity oxygen fuel (HVOF) gun from Sulzer Metco has been used in order to act as thermal barrier coating. Al_2O_3 and Ni-5Mo-5Al powders with 25 and 100 μm mean diameter respectively have been used to overcoat the Nimonic 80 substrates (10 mm x 20 mm x 2 mm) deposited previously with Re, Re30Cr10Ni and Ni20Al multilayer coating by TVA. The used fuel gas was hydrogen, the plasma jet temperature being higher than 5000 K. The coating consisted of a duplex - layer structure, an inner sigma (Re-Cr) or Re(Cr) layer and an outer alpha Cr(Al) or beta NiAl layer. The surface cluster morphology microstructures have been examined by SEM, finding almost spherical splats of Al_2O_3 powder very well melted and lenticular splats of the Ni-5Mo-5Al layer.

All samples were oxidized in a 5sccm air flow at 1373 K (300 K/h increasing and decreasing temperature rate) for up to 3.6 ks at the highest temperature showing very good oxidation resistance.

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