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Surface treatments in fluidized bed reactors

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Heat treatments of alloys in fluidized bed reactors have been carried out for more than twentyfive years. More recently this technology has been used for carrying out nitriding, carburizing and similar surface treatments. This technology offers certain advantages over other traditional methods for Surface Engineering. These advantages include a more precise process control, greater flexibility and more efficient mass and heat transfer control during the process. In this paper we present a design of a fluidized bed reactor capable for carrying out single, and multilayer surface coatings. *© 2000 Kluwer Academic Publishers*

1. Introduction

The fluidized bed (F.B) phenomenon is the phenomenon in which a bed of particles e.g. Al_2O_3, behave like a liquid, when a moving gas is fed through the bed. Some of the parameters that affect the quality of fluidization in a F.B reactor are the properties of solids and fluids used, the bed geometry, the gas flow rate, the type of gas distributor and the reactor design.

Surface treatments that have been developed using F.B technology include nitriding, carburizing, nitrocarburizing B, Cr, Al, Si, V and Ti containing coatings, and the deposition of hard carbide and nitride layers [1–16].

Multielement diffusion coatings have been achieved by researcher [17], by processes other than F.B technology. These multielement coatings, exhibit improved oxidation and corrosion properties, and are widely used in many industrial applications. In this paper we present a simple method for obtaining single, multielement and hard coatings using F.B. technology.

2. Experimental procedure

The F.B reactor used is shown schematically in Fig. 1. The main parts of the reactor included:

- The F.B reactor unit
- The gas preheating system
- The furnace for heating the reactor
- The control panels and measuring instruments
- The reactants feeding system
- The trapping of hazardous substances unit

The reactor material was 310 steel and the F.B reactor distributor contained 300 holes of 1,5 mm diameter giving a 5.8% of open space. The system included a powder feeding mechanism, which allowed the continuous feeding of reactive powder substances to the reactor, throughout the treatment period. The inert fluidizing material was Al_2O_3 powder of $\approx 100\ \mu m$ diameter. Inert Argon gas was used as a fluidising gas at a rate between 0.6–0.7 m^3/h. All the remaining reactants required for the specific coating deposition were either placed in the F.B reactor at the beginning of the experiment, or fed through the continuous feeding unit at a constant rate.

This experimental set up is simple and flexible and allowed to deposit a wide range of single and multielement coatings. The substrate materials used were steels Ni and Ti alloys and the coatings deposited were Cr, Al, Cr/Al, Cr with rare earth elements (Yt or Hf), and Cr/Al/Yt. The deposition temperature was between 1000–1100°C and the deposition time varied from 1–3 hrs. In Table I we summarize the coating conditions used and the type of obtained coatings.

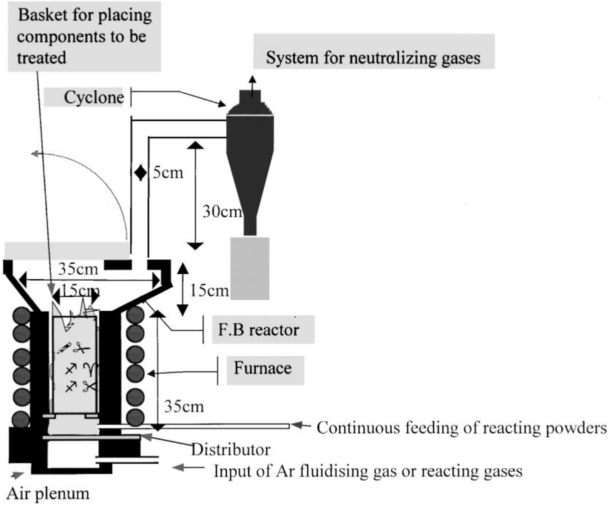

Figure 1 Schematic representation of the fluidised bed reactor.

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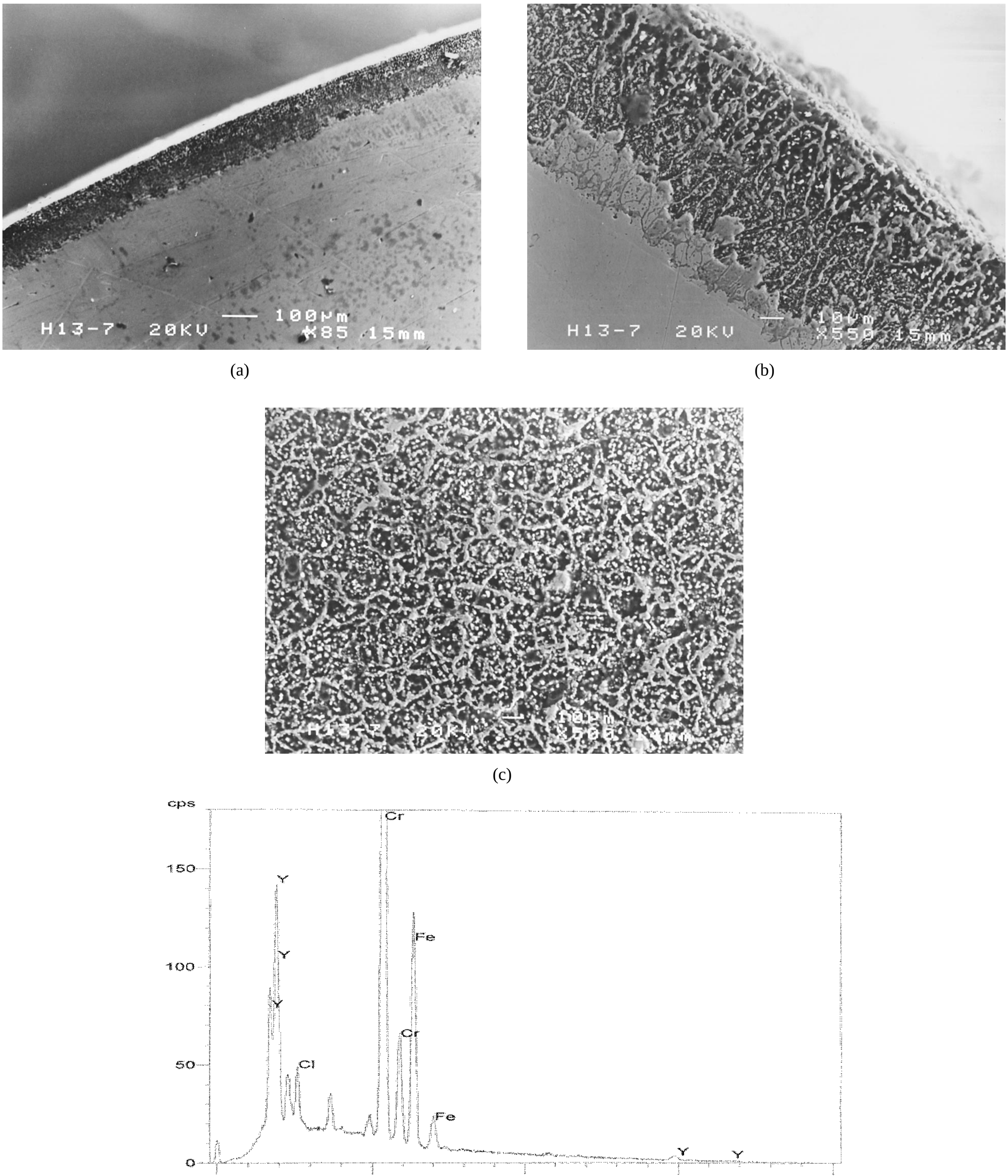


(a) (b)

(c)

(d)

Figure 2 (a) Typical morphology of Cr-Yt coating on H13 steel; (b) Typical morphology of Cr-Yt coating on H13 steel; (c) Surface morphology of Cr-Yt coating on H13 steel; (d) Qualitative chemical analysis of Cr-Yt coating.

TABLE I Coatings depositions parameters

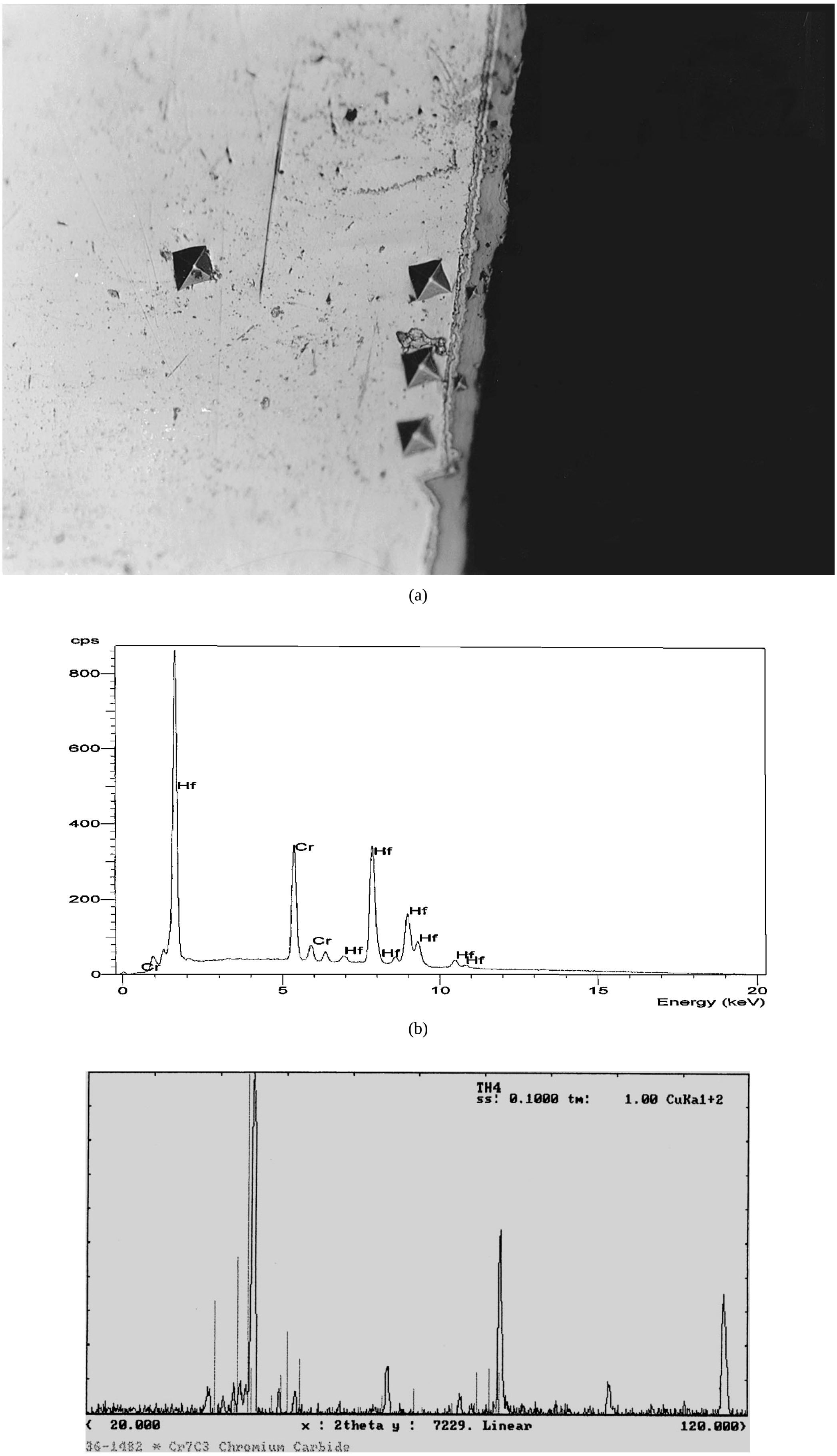
Coating	Compounds used	Temperature °C
Cr	NH4Cl, Cr	1000
Cr Hf	Cr, Hf,* HfCl4,* NH_4Cl*	1000
Al Hf	AlCl3,* $HfCl_4$*	1050
Cr Al	Fe - Cr - Al - C, NH_4Cl*	1050
Cr Yt	Yt, $CrCl_3$*	1100
Cr Al Yt	Fe - Cr - Al - C, Yt, NH_4Cl,*	1050
Cr Al Yt	Yt, $CrCl_3$,* $AlCl_3$*	1100

*This compound is fed through the powder feeding system.

3. Results and discussion

The type of coatings obtained in the F.B reactor and their specific morphological characteristics are given in Table II. Fe and Ni based alloys are the principal materials used in high temperature applications, such as utility boilers and gas turbines, where resistance to high temperature oxidation and corrosion is required.

Certain elements, such as Yt, Hf, La, Ce etc, when added in small quantities either in the bulk alloy or in the surface, have been found to have beneficial effects in high temperature oxidation and thermal fatigue



(a)

(b)

(c)

Figure 3 (a) Microhardness markings on the Cr(Hf) Carbide layers (×200); (b) EPMA results from the Cr(Hf) Carbide layers obtained on H13 steel; (c) X-Rays diffraction results of the Cr(Hf) Carbide layer obtained on H13 steel. Carbide peaks and the sample spectrum.

TABLE II Substrate materials and coatings prepared in the F.B. reactor

MATERIAL	COATING/LAYER	THICKNESS	COMMENTS
H13 steel	Chromium (Hf) Carbide	25 μ–30 μ	Very good adherence
602 alloy	Al (Hf traces)	Variable thickness 30 μ–100 μ	Very good adherence - Soft coating
INCONEL 718	Al		Non adherent coating
Ni-Al-Ti alloys	Cr / Hf	10 μ–15 μ	Non uniform deposition
Ti-Al-V alloys	Cr (Hf traces)	20 μ–25 μ	Non uniform deposition
10CrMo910	CrYt	150–200	
H13 steel	CrYt	150–200	
10CrMo910	CrAlYt	150–200	Very good adherence and uniformity in all coatings
RENE 80	CrAl	150–200	
INCONEL 718	CrHf	15–20	

properties of these alloys. This effect which has become known as reactive element effect, has been well documented [17] and has led many researchers to attempt to introduce simultaneously Cr Al and Yt, or Hf into the surface of Fe and Ni base alloys.

Many of the multielement coatings obtained on the Fe and Ni alloys shown in Table II exhibited very good adherence. The deposition of a R.E. element together with Cr / Al is a relatively simple task using the F.B reactor. In Fig. 2a–d, typical morphologies of the Cr / Yt coating obtained on H13 steel are shown, as well as a qualitative chemical analysis of the coating. This coating had average thickness of 150–200 μm and was obtained only after two hours of treatment. This type of coating is expected to have excellent oxidation and thermal fatigue properties.

A Cr(Hf) carbide coating deposited on H13 steel is shown in Fig. 3, together with the microhardness indentation markings on the substrate and coating, as well as the qualitative chemical analysis results of the coating. This coating of 25 μm thickness was obtained after 2 hours of treatment and was characterized by very good adherence and Vickers microhardness values between 1533–1971 Hv. The X-rays diffraction results indicated as a possible carbide the Cr_7C_3, see Fig. 3c. The exact form Hf is present in this coating has not been clarified beyond doubt.

The good quality of coatings, their good adherence, together with the flexibility and the simplicity of the F.B method make it very attractive for further development.

4. Conclusions

The F.B. technology has been successfully used to deposit good quality multielement coatings containing R.E. elements and Cr/Al on Fe and Ni base alloys, as well on hard carbide coatings of reasonable thickness on Fe alloys. The method is simple, flexible, environmentally friendly, and should be considered as a serious alternative to similar Surface Engineering processes.

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