

# Effect of Heat Exposure on Grain Growth and Oxidation Behavior of Cobalt Based Composite Powders by Facile Suspension Synthesis



N F Kadir, A Manap, M Satgunam, N Afandi, S Mahalingam, S M Yunus

**Abstract:** In this study the intermetallic-matrix composite powders  $\text{CoNiCrAlY-2wt. \%Al}_2\text{O}_3$  were characterized in order to investigate the effect of heat exposure time on morphologies, grain growth and phases formed by facile suspension route synthesis. The as-synthesized powders were examined by Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDX) and X-ray diffraction (XRD). The formation of NiAl phase was noticed after 1 hour of heat treatment. The average particle size of intermetallic-matrix composite powders  $\text{CoNiCrAlY-2wt. \%Al}_2\text{O}_3$  increased as the heat exposure time increased. It is found that the reinforcement of alumina allowed the particles to uniformly distributed when the sample was heated for 10 hours. The formation of NiAl started when the sample was heated at 1 hour and NiAl continued to form when heated at 10 hours.

**Keywords:** Composite powders, Heat and Oxidation

## I. INTRODUCTION

Currently, many attempts are conducted to improve efficiency of the advanced gas turbine system as such increasing inlet operating temperature. It is important to perform extensive research for efficient gas turbine engines with more reliable, cost-saving and longer lifetime of coating. This is to control total  $\text{CO}_2$  emission and sustain healthier environment in human live. Thermal barrier coating (TBC) is predominantly applied onto metallic substrate to act as protective barrier from mechanical and thermal failures.

Researchers had focused on improvement and development of super alloys as it has high strength, good thermal stability and be able to protect coatings from rapid oxidation and corrosion. However, as these super alloys expose to extreme environments, they are at high risk towards severe corrosion and oxidation failures. TBC system consists of metallic substrate, metallic bond coat (usually  $\text{MCoCrAlY}$ ,  $\text{M} = \text{Co}$  or  $\text{Ni}$  or  $\text{CoNi}$ ), and ceramic top coat. During service, a layer will form namely Thermally-Grown Oxide (TGO) between top coat and bond coat interface. However, rapid oxidation leads the formed TGO layer to spallation and delamination of coating. Thus, bond coat powder preparation plays crucial role as the initial oxide content of the powder should be restricted and later deposition technique stage can result in an improvement of oxidation behavior with desirable TGO properties. One of the remarkable advantages of Cobalt based super alloys is that it has a major effect especially on creep strength [1]. In addition, creep resistance and oxidation resistance at elevated temperatures can be significantly improved by addition of dispersoid, namely alumina as this dispersoid will enhance the TBC lifetime when it is doped with bond coat alloy powders [2, 3]. The TGO properties can be modified by means chemical composition and microstructure of the bond coat powder [4-6]. Many past works have focused to intermetallic compound such as Unocic et. al [7] concluded that 2wt. % addition of alumina in  $\text{CoNiCrAlY}$  bond coat powder gave the lowest mass gains during oxidation and best scale adhesion of coating. Other than that, this oxide dispersion strengthening is one of feasible approaches to improve room temperature ductility of a material by means grain refinement [10, 11].

From past work,  $\text{NiCrAlY}$  alloy was mixed via a suspension route in order to achieve a uniform distribution of  $\text{Al}_2\text{O}_3$  submicron particles [8]. However, for  $\text{CoNiCrAlY}$  alloy there is no feasibility study by suspension route with 2 wt.%  $\text{Al}_2\text{O}_3$  had been done. Conventional methods such as mechanical alloying and combustion synthesis are prone to chemical contamination and gives relatively high level of porosity which contribute to mechanical failures [9-11]. Hence, facile suspension route is believed to be low cost and simple tooling procedures to control particle size, minimal contamination and obtain homogenous distribution of composite powder feedstock [5]. Hence, in this research work,  $\text{CoNiCrAlY}$  alloy with addition of 2 wt.%  $\text{Al}_2\text{O}_3$  powder by facile suspension route was prepared. Samples were heated at  $1000^\circ\text{C}$  and characterized with variation of heat exposure time (0 hour, 1 hour, 10 hours).

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## II. RESEARCH METHODOLOGY

The powders used in this study are gas atomized CoNiCrAlY (AMDRY 9951, Sulzer Metco) and aluminium oxide ( $\text{Al}_2\text{O}_3$ ). 2wt. % addition of alumina is poured into beaker containing  $\text{C}_2\text{H}_5\text{OH}$  solution as suspension medium and stirred. Next, CoNiCrAlY powder was dissolved in the solution. This mixing process will take about half an hour. The slurry solution was wet milled for one hour and oven dried for 24 hours. A thermal exposure test was conducted at three different hours; 0 hour, 1 hour, and 10 hours at 1000 °C to investigate the microstructures growth and phases formed after heat exposure. Both quantitative and qualitative measurements were performed. Heated powders were then characterized with Scanning Electron Microscope (SEM) to observe morphology and particle size at magnification of 1k, Energy Dispersive X-ray (EDX) to observe elemental composition in the heated samples and X-Ray Diffraction (XRD) for phase identification. The XRD analyses were carried out with a Philips diffractometer with  $\text{CuK}\alpha$  radiation (40 kV, 30 mA).

## III. RESULTS AND DISCUSSIONS

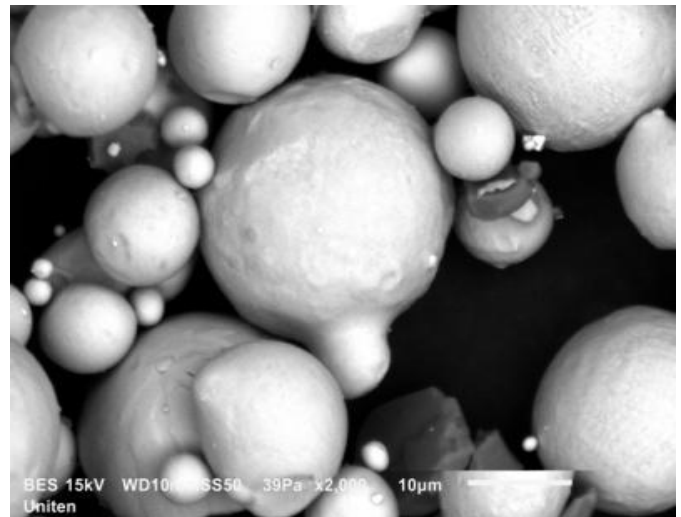
### Results

#### Grain growth

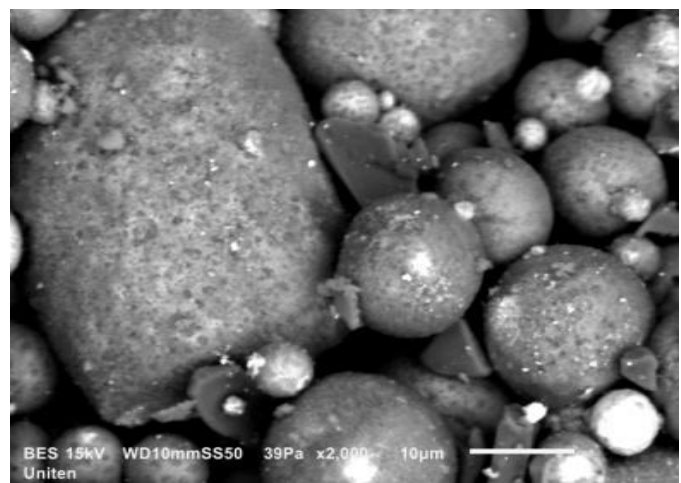
Figure 1 shows SEM images of 3 samples; 0 hour, 1-hour and 10-hours under 1000°C in tube furnace. For 0-hr sample, CoNiCrAlY and  $\text{Al}_2\text{O}_3$  particles exhibit no changes in their morphologies. For 1-hr sample, the particles start to undergo pinning effect among the particles [12]. For 10-hr sample, the added alumina is diffused into the major alloy particle surfaces. Microstructures of the 3 samples are significantly different; from coarse particles to finely dispersed alumina on CoNiCrAlY particle surfaces. In terms of particle size, as the heat exposure time increases, the particle size also increases. Average particle size calculation is done using line intercept method (ASTM E112-13). The average of particles sizes was calculated for 0 hour sample, 1 hour sample and 10 hours sample are 7.761  $\mu\text{m}$ , 8.218  $\mu\text{m}$  and 8.480  $\mu\text{m}$ , respectively using imageJ software. The heat-treated samples are believed to undergo grain growth due to an increase in bonding mechanism at the particles interface by material diffusion [12]. Hence, it is believed that desirable heat treatment can promote for metallurgical bonding to a certain level which by this property mainly plays its role when protective coating under operation in severe environment [13].

#### Thermal oxidation test

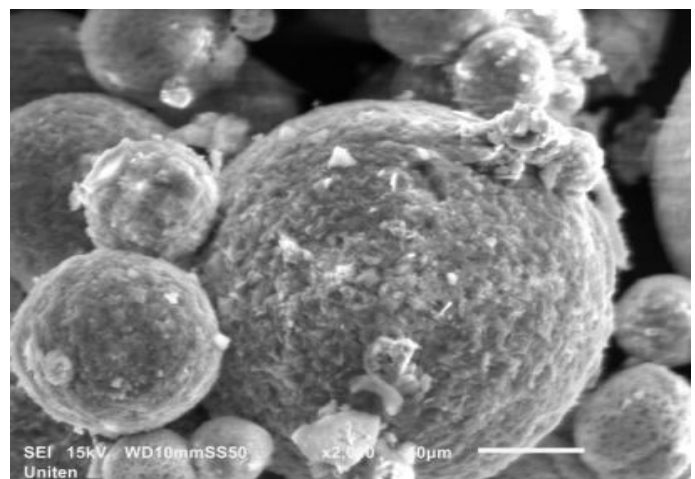
After 1 hour of heat exposure, from Fig. 3, it can be seen the peak intensities of powders have increased and the formation of NiAl phase was noticed throughout the heat treatment process. However, at 10 hour sample, the peak intensities are decreased because fine alumina particles have embedded into CoNiCrAlY particle surfaces. This may occur as the particles received heat activation energy for further agglomeration and bonding. At 10 hours sample the total oxide formed is increased during 10 hours of heat treatment and the particles had pinning effect between particles. However, this element composition changes are further confirmed with phase's identification.



(a)

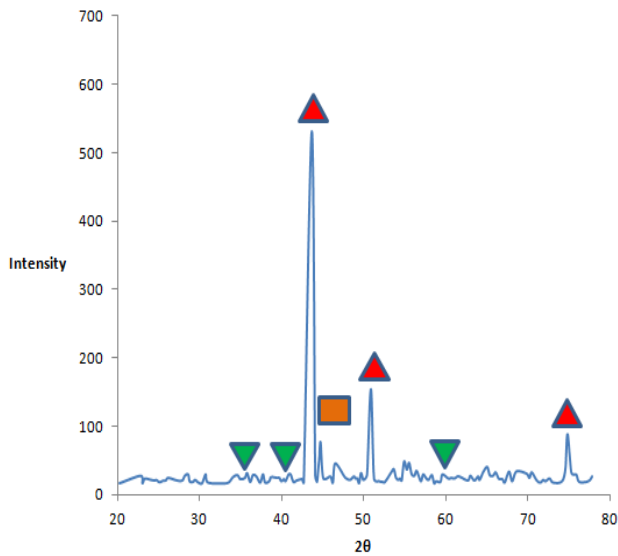


(b)

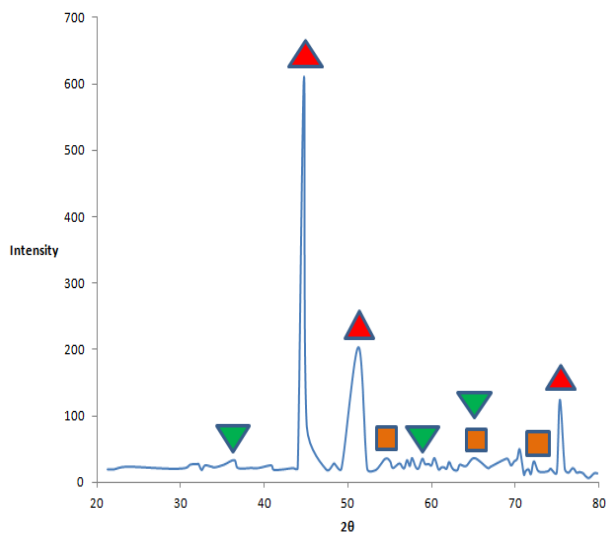


(c)

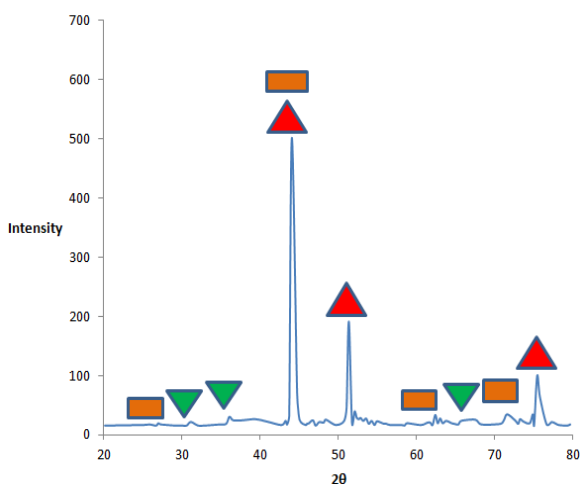
Fig. 1 SEM images for characterized samples a) 0 hour sample b) 1 hour sample c) 10 hours sample



(a)

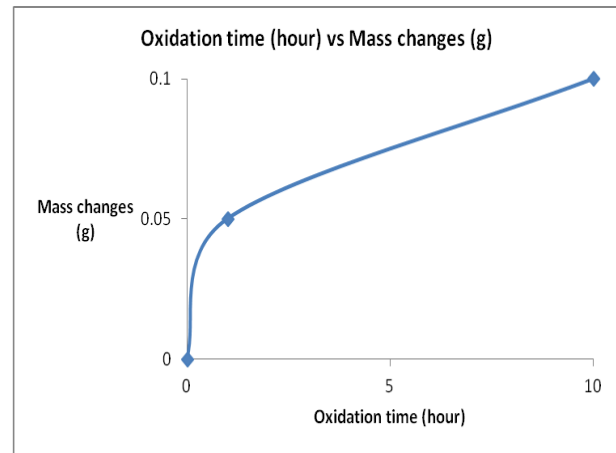


(b)



(c)

**Fig. 2 X-ray diffraction patterns of CoNiCrAlY-2wt %Al<sub>2</sub>O<sub>3</sub> samples at a) 0 hour b) 1 hour c) 10 hours**



**Fig. 3 Graph between oxidation time and mass changes of the as-synthesized and heat treated composite powders**

#### IV. CONCLUSIONS

From microstructure characterization and thermal oxidation test, it can be deduced that a homogenous oxide-dispersion CoNiCrAlY composite was obtained at 10 hours of heat treatment. It is shown that increase in heat exposure time resulted in uniform distribution of alumina reinforcements in the  $\gamma$  phase matrix but it is unable to retard for grain growth.

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