



## Microstructural evolution of mechanically alloyed Ni-based alloys under high temperature oxidation



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### ABSTRACT

To further improve the cost-benefit of MCrAlX (X is a reactive element like Y, Hf, Ta, etc.) alloys, the microhardness, microstructure and oxidation behavior of two Ni-based alloys, Ni<sub>60</sub>Co<sub>30</sub>Al<sub>10</sub> and Ni<sub>60</sub>Co<sub>10</sub>Cr<sub>20</sub>Al<sub>10</sub> without reactive elements were studied by scanning electron microscopy, X-ray diffraction and microhardness Vickers. These alloys were synthesized by mechanical alloying in a high energy ball mill and conventional sintering at 1100 °C under inert atmosphere. Milling time and Cr additions have a significant effect on microhardness of as-milled and sintered samples. Bulk samples were oxidized at 1000 °C for different times to analyze the possible growth of protective oxide scale, as well as the effect of Cr addition on the chemical composition of the oxide scale. After 3 h of oxidation the formation of Al<sub>2</sub>O<sub>3</sub> oxide scale in the Ni<sub>60</sub>Co<sub>30</sub>Al<sub>10</sub> alloy is evident. By reducing the Co content and incorporating 20 wt.% of Cr, the formation of an Al<sub>2</sub>O<sub>3</sub> oxide scale with the presence of Cr<sub>2</sub>O<sub>3</sub> was observed. Even the formation of homogeneous, continuous and protective oxide products in the surface of samples is evident, the microstructural changes after oxidation treatments have a significant effect on the decrease in microhardness of both alloys.

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### 1. Introduction

The development of more efficient gas turbine engines is the result of continuous improvements in several areas of engineering such as the design of turbines, combustion systems and new materials [1,2]. This last includes: fabrication of more resistant materials to creep and oxidation [2], advances in melt and casting technology [3] and the development of coating technology (thermal barrier coatings). Superalloys used as substrate materials for turbine blades are metallic materials for use at high temperatures. The main role of the substrate is withstanding the mechanical stress generated during use, while coatings are a very important part of advanced materials system to protect the turbine blades from oxidation and corrosion during service.

A thermal barrier coating (TBC) allows the thermal insulation for the turbine components with additional protection from corrosion and oxidation. The actual “state of the art” of TBC typically consists of a top coat Y<sub>2</sub>O<sub>3</sub>-stabilized ZrO<sub>2</sub> (YSZ) and an underlying metallic bond coat (BC) type MCrAlX (where M represents one or more elements such as Co, Ni, and Fe; and X is a reactive element added in minor proportion, which may be yttrium, hafnium, etc., or a mixture thereof) [4]. The BC must be able to provide corrosion resistance by the formation of a thermally grown oxide (TGO). The TGO must be consisting of a stable phase for the adhesion of the ceramic layer of thermal insulation [5,6]. Nickel based superalloys like NiCrAlX and/or NiCoCrAlX, have emerged

as alternative bond coats [7] due to their high chemical compatibility with the superalloy used as substrate, that allows a good adhesion substrate-coating, and to their compatibility in mechanical properties. The bond coat is probably the most important component in TBC systems. Its chemistry and microstructure have an important influence over the durability of the system, through the structure and morphology of TGO. The oxidation resistance of BC depends on the ability of the alloy to produce a continuous TGO, stable, slow-growing and adherent to the substrate [8]. The formation of α-Al<sub>2</sub>O<sub>3</sub> as a protective coating on the surface of a BC is the most desirable, not only for the low diffusion rate of oxygen and metal ions through it, but also for its high chemical and thermal stability [9]. However, the oxidation of a BC is usually accompanied by a rapid growth of non-protective oxides such as (Co, Ni)(Al, Cr)<sub>2</sub>O<sub>4</sub> and (Co, Ni)O [10].

On the other hand, mechanical alloying (MA) is known as a rapid processing route for the production of a wide range of dispersion strengthened structures, nanocrystalline materials and advanced alloys [11]. Even though, the processing is apparently simple, a group of new materials are produced by high-energy ball milling from elemental precursors in powder form. Prepared systems show a significant increment on their mechanical and physical performance based mainly on their high structural refining and homogeneous composition. Hence, a substantial control over the structure of alloys produced in laboratory conditions has been achieved. Most of the commercial powders used for the production of coatings are fabricated by gas atomization. So, one challenge in the field of powder alloys used as coatings is the application of high-tech processing techniques to produce high

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performance raw materials for bond coats, where a precise control of structure, morphology, crystal size and homogeneity is mandatory [12].

The present investigation deals with the structural, microstructural and microhardness characterization of two Ni-based alloys,  $\text{Ni}_{60}\text{Co}_{30}\text{Al}_{10}$  and  $\text{Ni}_{60}\text{Co}_{10}\text{Cr}_{20}\text{Al}_{10}$ , produced by mechanical alloying and conventional sintering; as well as, their oxidation behavior at relative high temperature. The aim of this investigation is to evaluate the response of these alloys to oxidation at high temperature as a function of their chemical composition, i.e., the Co and Cr content and the absence of reactive elements. The results of this study will act as an indicator of possible candidates for bond coats in terms of their chemical composition according to their capability to form protective oxide scales after oxidation.

## 2. Material and methods

Elemental powders of Ni (purity 99.9%, – 325 mesh), Co (99.5%, – 325 mesh), Al (99.9%, – 325 mesh) and Cr (99.9%, – 325 mesh) from Alfa Aesar were used as raw materials. The  $\text{Ni}_{60}\text{Co}_{30}\text{Al}_{10}$  and  $\text{Ni}_{60}\text{Co}_{10}\text{Cr}_{20}\text{Al}_{10}$  powder mixtures were prepared by combining different concentrations of the elemental powders. Prior to milling, weighted powder mixtures were blended for 30 min to obtain a homogenous mixture of powders. Millings were performed in a high energy SPEX (8000M) ball mill and hardened steel vial and grinding media were used. Two different sizes of ball mill were used, 1/2 and 7/16 in in diameter, three balls of each size. To minimize oxidation of the powder mixture, the steel vial was first evacuated with a vacuum pump, and then filled with argon and the milling times were set to 5 and 20 h; with cycles of 1 h of milling time and 0.5 h rest each to avoid the local temperature rise inside the vials during milling. The ball-to-powder weight ratio for all the experiments was 5:1 and methanol (3.7 wt.%) was used as process control agent. The powders were cold compacted in a uniaxial hydraulic press under a pressure of 1.5 GPa and sintered in sealed quartz ampoule under vacuum at 1100 °C during 3 h, with a heating rate of 5 °C/min. After sintering, the sample surfaces were ground to remove the typical oxide layer and were tested for oxidation at 1000 °C in air for 1 and 3 h. The structural evolution of the samples as well as the phase identification were followed by X-ray diffraction (XRD) in a Panalytical X'pert diffractometer using a Cu cathode ( $\lambda = 0.15406$  nm). The studies were performed in the  $2\theta$  range of 20–110°. The step size and acquisition time were 0.05° and 100 s, respectively. The crystallite size in as-milled samples was calculated from Sherrer's equation after eliminating the instrumental broadening and strain contribution. Microstructural characterization and chemical microanalyses were performed in a JSM-5800LV scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS) operated at 20 kV. Vickers microhardness tests were measured under 50 g load with 10 s of dwell time for powder samples, and 200 g load with 10 s for bulk samples, using a LM 300AT Leco MicroHardness tester. The hardness reported is the average of 10 measurements. For comparison, a commercial powder alloy AMDRY 997 (gas atomized prealloyed material supplied by Sulzer Metco) was also studied following the same characterization route of the mechanically alloyed (MA'ed) powders.

## 3. Results and discussion

### 3.1. Powder samples

Because of the milling process employed and the ductility differences in raw material, the final composition is different from the nominal composition. Variations in composition in milled products are due to mechanical drag and adhesion to milling media, mainly. Table 1 shows the nominal and chemical compositions from semiquantitative microanalyses on synthesized and commercial samples. The sample identification is included, and for synthesized samples the identification is related to the milling time. The presence of oxygen was not detected in milled products. This indicates that the oxidation level was very low. The most important variation was observed in aluminum content, this metal is the most ductile and probably the adhesion to milling media was stronger than in other metals.

The AMDRY 997 (A997) alloy is a Ni-based alloy that is used in the aerospace industry as a BC in turbine blades [13–15]. A997 constituents is similar to those selected in this work; the main difference with the studied alloys is the Tantalum and Yttrium content. Y is very reactive with oxygen; however, the addition of this element is due to the improvement of TGO scale adherence, increases of the creep strength of the TGO and the TBC life is extended when they are on bond coats with oxygen reactive elements [3]. On the contrary, Ta is used to increase the oxidation resistance; the use of both elements increases the life of BC. These elements are more expensive and difficult to handle in powder metallurgy process. This research aims to characterize MA'ed metallic alloys to determine if they can be potential candidates for use as BC, without the need to add a reactive element to form a high quality TGO.

In powder metallurgy it is very important to know the nature of powder particles to understand the effect of processing. Scanning electron microscopy (SEM) is one of the most useful techniques for observing the discrete characteristics of metal powder particles. Fig. 1 shows the morphology and size of the elemental powders used in this work. As can be noticed, the morphology and size of the raw materials is quite different. Particle size is one of the most important characteristics in powder metallurgy. It can be determined by several techniques making often the assumption of a spherical particle shape. From a projected image of a rounded but irregular shape, six possible measures of particle size can be obtained, three based on projected dimensions and three based on equivalent spherical diameters [16]. Through SEM analysis the properties of shape, size, microstructure and chemistry of individual powder particles were determined. The particle size was obtained by measurements of the projected width of single particles observed in SE-SEM images. The Ni powders present the smallest and bimodal particle size ranging from 10–25  $\mu\text{m}$ , while those of Co show an irregular morphology with larger particle size in the range of 30–50  $\mu\text{m}$ . For instance, the powders of Al seem to exhibit a bimodal particle size distribution and rounded morphology. Finally, the morphology of Cr powders consists in flake-like particles with the largest particle size among the elemental powders.

**Table 1**  
Nominal\* and chemical composition by EDS–SEM of the powder alloys (wt.%).

Alloy	Milling time (h)	Sample	Ni	Co	Cr	Al	Y	Ta
$\text{Ni}_{60}\text{Co}_{30}\text{Al}_{10}$	0	0A	63*	32*		5*		
	5	5A	64	30		6		
	20	20A	63	32		5		
$\text{Ni}_{60}\text{Co}_{10}\text{Cr}_{20}\text{Al}_{10}$	0	0B	65*	11*	19*	5*		
	5	5B	64	12	18	6		
	20	20B	66	14	15	5		
AMDRY 997		A997	Bal.	20–26*	18–23*	6–11*	0.3–0.9*	2–6*
$\text{Ni}_{43.9}\text{Co}_{23}\text{Cr}_{20}\text{Al}_{8.5}\text{Ta}_4\text{Y}_{0.6}$			42	23	19	9	ND	7

ND – not detected

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