

# The synthesis of NiCrAlHf powders with mechanical alloying method for thermal sprayed coating

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**Abstract.** NiCrAl is a material commonly used for coating in order to increase corrosion or oxidation resistance of materials applied at high temperatures. The addition of hafnium (Hf) as reactive elements to NiCrAl coating are found to prolong the life of the industrial gas turbine blade and increase its performance. NiCrAlHf powders used as the base material were mixed using planetary mill device for 36 hours with a speed of 25 Hz or 1500 rpm. Characterization of NiCrAlHf powder was investigated before and after milling process using Scanning Electron Microscopy (SEM) and X-ray diffraction (XRD). The results showed that the synthesized powders were agglomerated. The obtained particle size of 9.64  $\mu\text{m}$ , 19.97  $\mu\text{m}$ , 39.27  $\mu\text{m}$ , 19.93  $\mu\text{m}$ , and 22.8  $\mu\text{m}$  was observed before milling process, after 9, 18, 27, and 36 hours milling process, respectively. The phase formed on the synthesized NiCrAlHf powder after 36 hours milling process included Ni and Cr with crystallite size of Ni 25.4 nm and Cr 8.8 nm calculated by Scherrer method. In addition the peak profile of the Al phase gets more amorphous after 27 hours.

## 1. Introduction

The NiCrAl coating is an interface layer for thermal barrier coating that systems commonly used to protect components made of superalloys for high temperature usage. In NiCrAl bond coat systems, reactive elements are required as stabilizers and enhancement for protective binding to extend the life of the gas turbine blade. The commonly used reactive elements are from rare earth element, such as hafnium, due to its high melting point and good corrosion resistance. In addition, hafnium is also primarily used to improve mechanical properties of the coating. Addition of reactive elements is also proven to decrease the formation of oxide scales, thus prolonging the lifetime of coating [1].

Thermal barrier coating that is composed with nano structured bonding has shown improved performance, especially at oxidation behavior at high temperature condition, compared with conventional micro structure bonding [2]. One of the known methods to form nano structured material is mechanical milling [3]. Ball milling as one of mechanical milling method has been proven to successfully prepare a variety of alloy powders exhibiting supersaturated solid solutions, quasi-crystals, amorphous phases and nano intermetallic compounds. The fundamental principle of size reduction in mechanical attrition devices lies in the energy parted to the sample during impacts between the milling media [4].



This study aims to introduce synthesis method of nano-crystallite NiCrAlHf powders through mechanical process and investigate the morphology of NiCrAlHf powders before and after milling process.

## 2. Experimental procedures

Nickel, chromium, aluminum and hafnium powders are weighed with composition as follows:

**Table 1.** Composition of powders (gram).

Element	Ni	Cr	Al	Hf
Mass of powders (gr)	137.2	48	14	0.8

The amount of reactive element used (Hf) was 0.4% of the overall mass or 0.8 gram, because the reactive element of  $\geq 1\%$  or  $\leq 0.1\%$  can accelerate the removal of the protective oxide layer when it's applied. The powders were mixed and mechanically processed with planetary ball mill (SFM-1 desk-top type) with a milling speed of 1500 rpm for 36 hours. Ball to powder ratio is specified into 2:1.

To obtain the effect of milling time to crystallite size, every 9 hours of milling, mechanically processed powders were sampled and characterized. Morphology of particles are identified using scanning electron microscope (JEOL JIB 4610f), while phases of particles are identified with X-ray diffractometer (Rigaku SmartLab) with a wavelength of CuK $\alpha$ . X-ray diffraction result then will be analyzed through Scherer's method to obtain the crystallite size.

## 3. Results and discussion

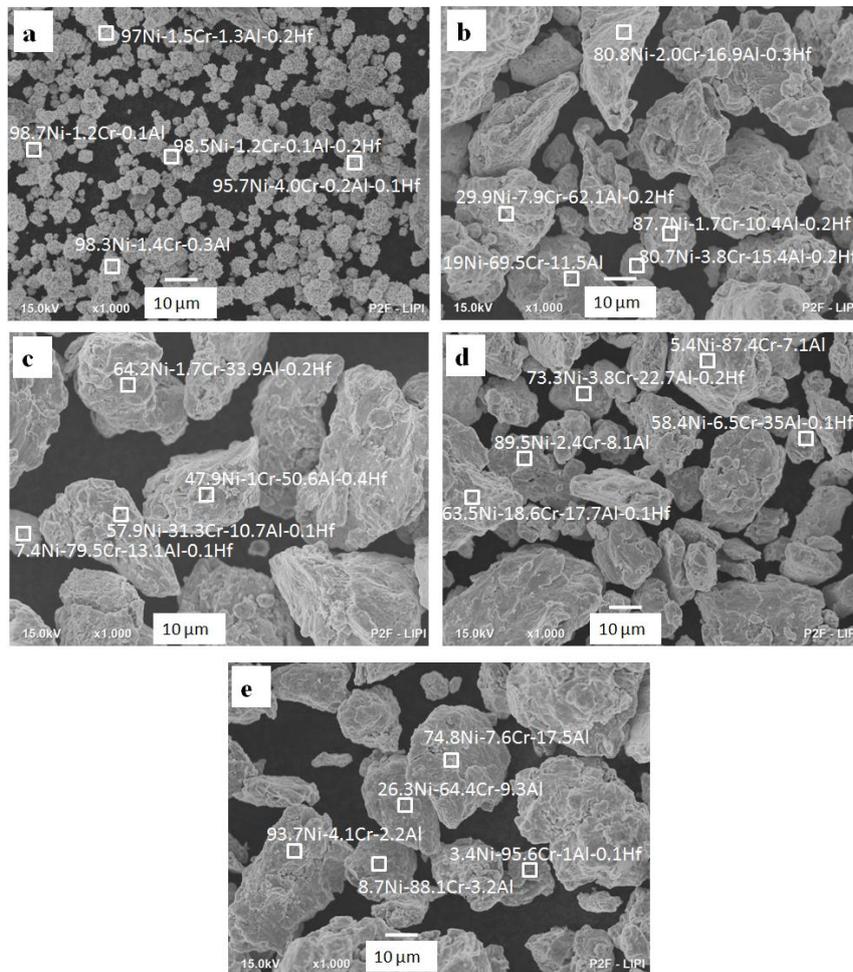
### 3.1 Scanning electron microscopy (SEM) characterization

Figure 1 shows the morphology of milled powder for 0 hour until 36 hours, with a step of 9 hours. From these images, an increase in particle size can be observed in accordance with milling time. At first the powders are observed to be in various shapes depending on the base powders with an average size of 9.64  $\mu\text{m}$ . After the first 9 hours of milling process, the powders agglomerated to form a homogenous shape and increase in size to 19.97  $\mu\text{m}$ . After 18 hours of milling, the agglomerated powders started to agglomerate further and gain an increase in size to 39.27  $\mu\text{m}$ . Agglomeration is caused as an effect of mechanical alloying when powders particles collide with enough energy to form interaction between the powders [5]. However, after 27 hours of milling the powders did not agglomerate further and having a breakdown to the size of 19.93  $\mu\text{m}$  and later increase in size again to 22.8  $\mu\text{m}$  after 36 hours of milling. This breakdown phenomenon may be caused by low binding energy of crystallites in the agglomerated form, therefore continued milling process cause the powder particles to be dispersed again. Figure 2 shows the measurement of particle size from NiCrAlHf powders using image-J software before and after milling process until 36 hours.

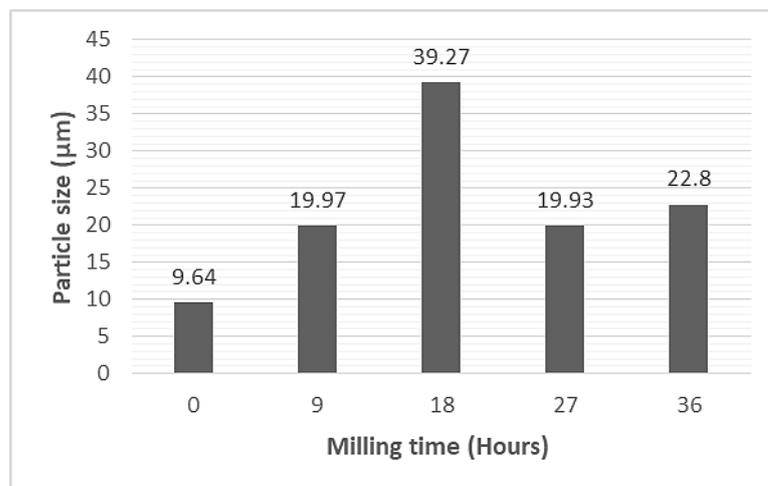
### 3.2 X-ray diffraction (XRD) characterization

Phase analysis of the sampled powder was characterized with X-ray diffraction and the results are shown in Figure 3. From the graph it can be observed that before milling process, phases of elementary powder used in this experiment are obtained. However, after milling process, a decrease in peak intensity of detected phases can be observed. It indicated that crystallite size was reduced.

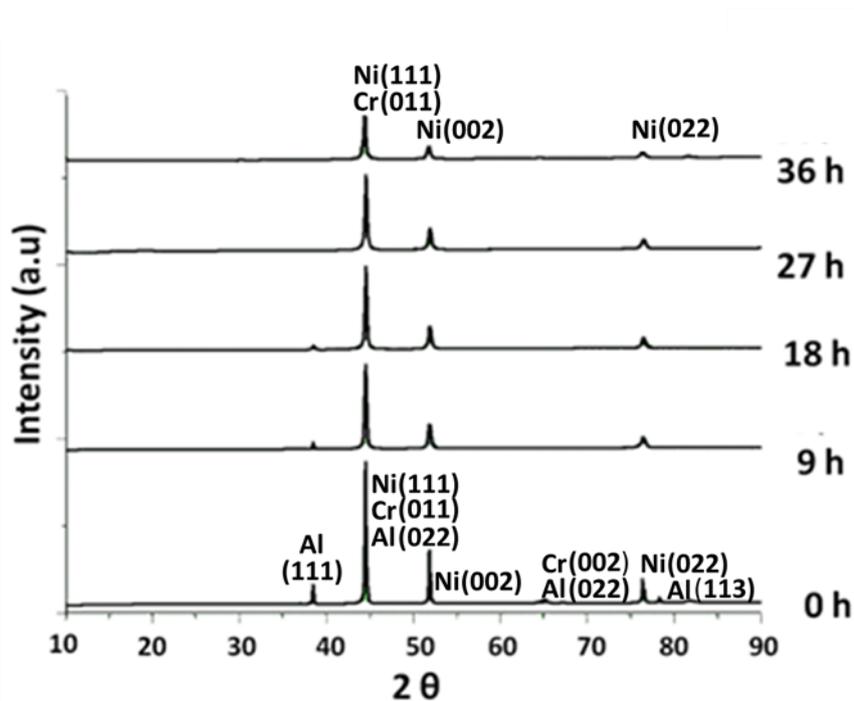
From the figure, it can also be observed that milling time plays an important part in the decrease of crystallite size and deform some of the material. Aluminum peak are not observed after 36 hours of milling time indicate that there is a change in the crystal shape that cause it to deform into an amorphous phase [6]. A study shows that mechanical alloying process with nickel and chromium presence cause by a deformation of crystal shape into amorphous [7, 8].



**Figure 1.** SEM image of NiCrAlHf powder (a) before milling, after milling process of (b) 9 h (C) 18 h, (d) 27 h, and (e) 36 h.



**Figure 2.** Graphic particle size of NiCrAlHf powders using image-J software at various milling times.



**Figure 3.** X-Ray diffraction pattern analysis of NiCrAlHf powder.

Further analysis of the XRD result with Scherrer's method was done to analyze the crystallite size of processed powder [9]. The method uses calculation shown in equation 1 to estimate crystallite size of powders.

$$D = K\lambda/B \cos \theta \quad (1)$$

Where:

D = Crystallite size (nm)

K = Constant (0,9)

$\lambda$  = X-Ray wavelength (nm)

B = FWHM at  $2\theta$  (rad)

Results of crystallite size calculation are shown in Table 2. According to the results it can be observed that mechanical process of the powders reduces crystallite size of powders, even though there is an increase in particles size due to agglomeration. Milling process duration caused the material size to be decreased further. It can also be observed that the crystallite size of powders is in the scale of nanometer, thus it should be ideally used as a starting material for coating process.

**Table 2.** The effect of milling times on crystallite size calculated by Scherer method.

Milling Times (hours)	Crystallite size (nm)		
	Ni	Cr	Al
0	61.3	28.5	79.1
9	30.8	13.3	54
18	31.9	12.0	37.6
27	28.1	9.2	
36	25.4	8.8	

#### 4. Conclusions

From the experiments there are several conclusions that could be drawn (i) Mechanical alloying process using planetary ball mill can be used to mix Ni-Cr-Al-Hf powders to an alloy for bond coat material; (ii) Milling process for 36 hours caused the powders to form agglomeration with an average size of 22.8  $\mu\text{m}$ ; (iii) After 36 hours of milling, phase identification shows that the alloyed powders are mainly constituted of nickel and chromium phases, while other phases are deformed into an amorphous shape; (iv) Using the Scherrer's method, a decrease in crystallite size was able to be proven after 36 hours of mechanical alloying process.

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