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Structural and morphological evaluation of 25% vol. Ni/Al_2O_3 nanocomposite powders produced by mechanical milling

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Abstract. The evolution of the Al_2O_3/Ni (25% vol. Ni) composite powders, during the milling and the stability of the composite phases were investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray microanalysis (EDX). SEM images show a high level of homogenization of the Ni and Al_2O_3 phases for milling times larger than 120 minutes. The X-ray study indicates no reaction between the two phases. The crystallite grain size decreases with the milling time for both phases.

Introduction

Ceramic particles are combined with metallic powders in order to improve some mechanical properties of ceramics. Ceramic matrix composites are designed for applications where metal matrix composites can't be used, presenting higher strength, stiffness, hardness, wear resistance and reduced coefficient of thermal expansion in comparison with the metal matrix composites [1, 2].

The Al_2O_3/Ni nanocomposite powders are employed in aerospace, naval, automotive industries and so on. The dispersing of nanoparticles as nickel improves the mechanical properties of alumina-based composites. A uniform dispersion of nickel nanoparticles enhances the mechanical properties of Al_2O_3–Ni nanocomposites [3, 4].

Alumina is used due to its high strength and relatively small coefficient of thermal expansion. The improvement of alumina ductility can be achieved using powders such as nickel [5, 6], magnesium [7], copper [8, 9], iron [10], molybdenum [11], titanium [12], and so on. High ductility, good thermal stability at high temperature, and high corrosion resistance make nickel a suitable choice for metal–ceramic composite elaboration. Nickel is a highly resistant to oxidation metal, presenting a high mechanical resistance at high temperatures [13].

This paper studied the obtaining and characterization of alumina and nickel nanocomposite powders by mechanical milling.

Materials and experimental procedure

Al_2O_3/Ni nanocomposite powders with 25% vol. Ni were obtained by high-energy mechanical milling starting from a mixture of Al_2O_3 and 123-carbonyl Ni commercially powders. The powders were mixed in a Turbula-type blender for 20 minutes and then milled in a Fritsch Pulverisette 4 planetary ball mill under argon atmosphere up to three hours. Samples were collected after 60, 120 and 180 minutes respectively. The used ball to powder ratio was 8.9:1. The parameters for the milling process were: ball diameter - 14 mm, main disk's rotational speed - 400 rpm, and vials rotational speed - 800 rpm.

The evolution of the powders morphology and composition during milling and the phase stability of the composite were investigated by X-ray diffraction (XRD), using a Schimadzu XRD-6000 diffractometer, operating with Cu-Kα radiation (λ=1.5406 Å), in the 2 theta range 20 - 100°. The crystallite mean size was calculated from the diffraction patterns using the Williamson – Hall method. In order to do so, the resolution of the diffractometer was determined from the diffraction pattern of a reference sample.

The particles size distribution has been determined using a Laser Particle Size Analyzer, Fritsch Analysette 22–Nanotec. The scanning electron microscopy (SEM) images were recorded on the JEOL JSM 5600 LV microscope, equipped with energy dispersive X-ray microanalysis spectrometer (EDX, INCA 200 software). The powders were embedded in resin and metallographically prepared for standard observation for the optical and microscopic studies.

Results and discussions

The particles size distribution of the powder having a nickel content of 25% vol. versus milling time is presented in Fig. 1.

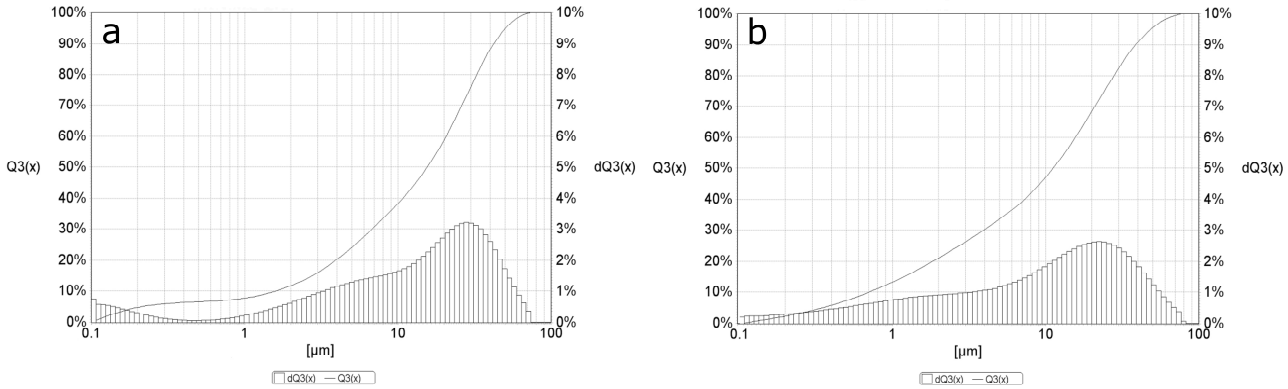


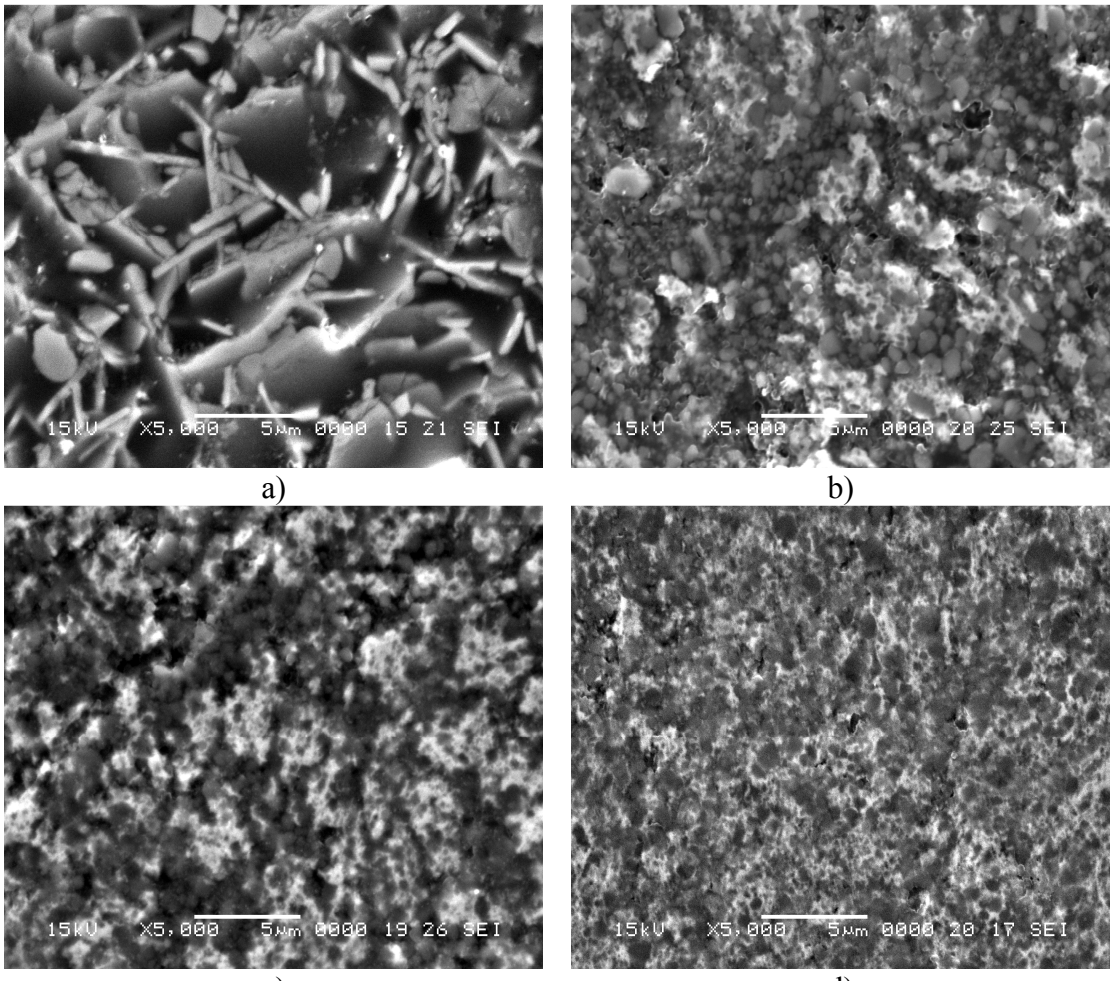
Fig. 1 Particle size distribution of the 25% vol. Ni/Al_2O_3 composite powder for the unmilled sample (a) and milled for 180 minutes (b).

The particle size distributions presented in figure 1 show a change in the particle size from the starting powder (before milling) to composite powder milled for 180 minutes. For the un-milled sample it is visible a large distribution of the sizes around 20 – 40 µm, corresponding to larger nickel particles surrounded by alumina particles. This maximum is accompanied by second maxima toward 2-7 µm corresponding to uncovered nickel particles (the used nickel particles are in this range). Finally to sizes lower than 100 nm a new maxima is visible (unfortunately at the equipment detection limit) attributed to the alumina particles. Milling the sample (Fig. 1 – b), the grain refinement is initiated, leading to elimination of the maxima corresponding to nickel and alumina single particles and the promotion of the nickel/alumina maxima to sizes nearby 10 – 15 µm. The evolution of the sizes presented in Fig. 1 is confirmed by computing the parameters of the particle size distribution, as presented in table 1.

Table 1. Particle size distribution parameters (D_{10}, D_{50}, D_{90}) of the 25% vol. Ni/Al_2O_3 composite powder, un-milled sample and milled for 180 minutes.

	0h MA	3h MA
D10 [µm]	1.59	0.70
D50 [µm]	15.53	11.17
D90 [µm]	52.30	38.73

The formation of the nanocomposite particles intuited by particle size distribution analysis is confirmed by SEM and it is studied as the milling progress from 0 to 180 minutes. SEM images show two types of particles for the sample that was not milled (Fig. 2a): large individual nickel particles and small elongated alumina particles dispersed on the surface of the larger nickel particles.



a) b)

c) d)

Fig. 2 SEM images for: a) Ni/Al_2O_3 starting mixture before milling, b) 25% vol. Ni/Al_2O_3 composite milled for 60 minutes, c) 25% vol. Ni/Al_2O_3 composite milled for 120 minutes, d) 25% vol. Ni/Al_2O_3 composite milled for 180 minutes.

By increasing the milling time up to 180 minutes, the powder has finer and more homogeneous particle dispersion (Fig. 2 b, c, d).

The shape of the particles mechanical milled for 180 minutes is dramatically changed by comparison with the particles of the starting mixture, leading to similar shaped particles for both nickel and alumina components.

It can be observed that increasing the milling time up to 180 minutes the shape of the particles gets more rounded and is difficult to distinguish individual nickel and alumina particles. Due to the higher ductility of the nickel compared with alumina the nickel particles cover the alumina particles, forming a metallic/ceramic nanocomposite material

The chemical homogeneity of the milled powders was studied by X-ray microanalysis and the elemental distribution maps are shown in Fig. 3.

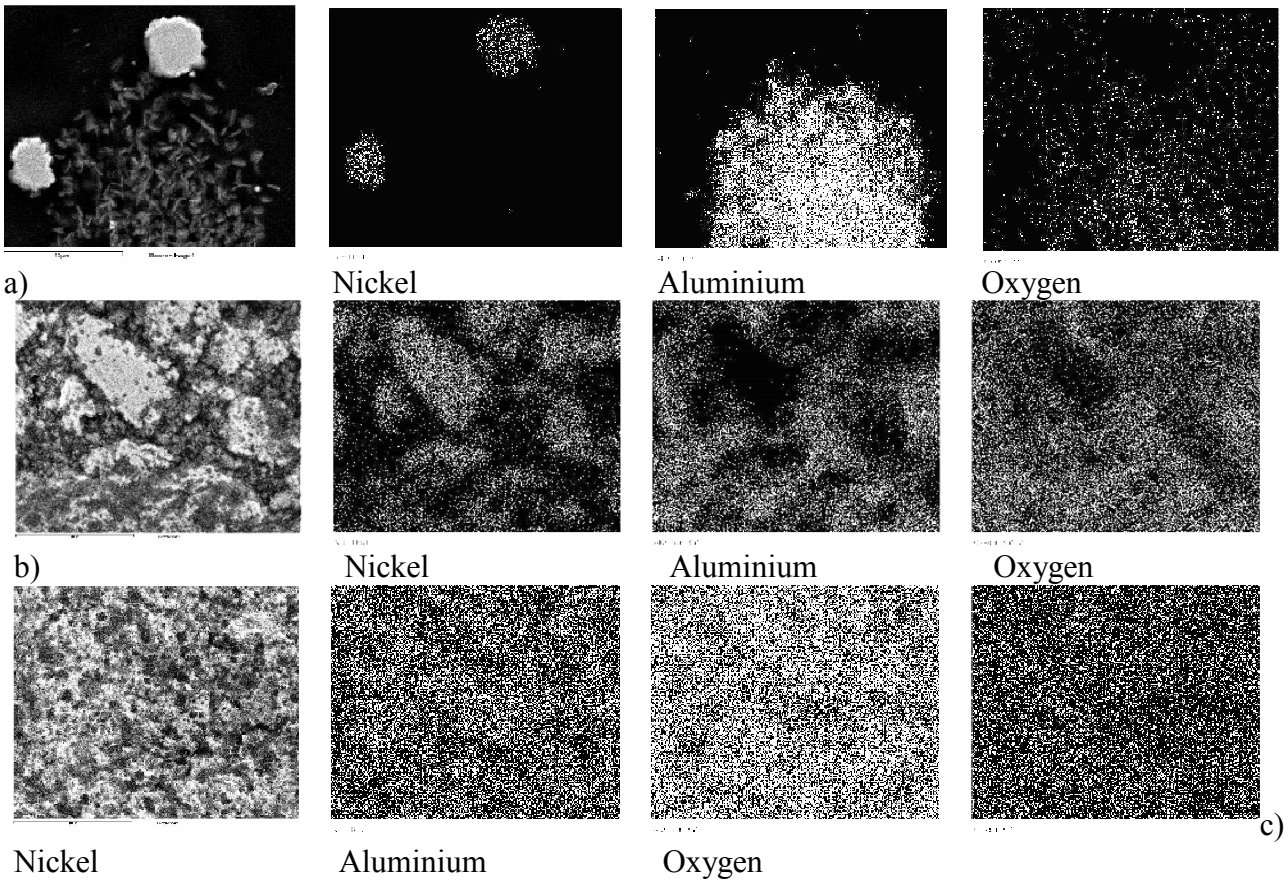


Fig. 3 Elemental distribution maps for 25% vol. Ni/Al_2O_3 composite powders milled for a) 0, b) 60 and c) 180 minutes respectively.

Looking at the distribution maps, we concluded that the elemental distributions of the Ni, Al and O in Al_2O_3/Ni particles are modified during the milling time. In the starting sample (Fig. 3a) are visible the large Ni particles near the large area with Al_2O_3 particles. Milling leads to the mixing of large Ni particles and Al_2O_3 particles. The EDX results for the powder mechanically milled for 60 minutes (Fig. 3b), show changes in the distribution of elements, but the alumina and nickel distributions are not uniform. After 180 minutes of milling (Fig. 3c) a good chemical homogeneity of the particle is observed and it is concluded that after this milling time, a Ni/Al2O3 composite is formed.

An elemental distribution on a line was done for the powder milled for 180 minutes, in order to observe the embedment of the Ni and Al_2O_3 particles in the composite particle. The linear EDX analysis of the powder milled for 180 minutes is given in Fig. 4.

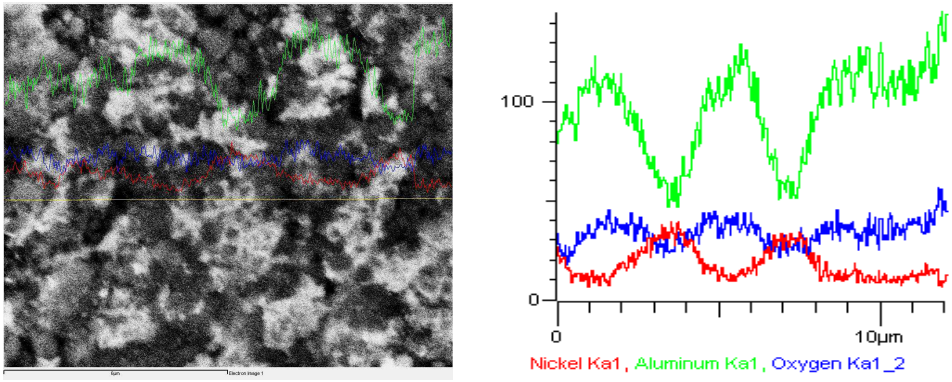


Fig. 4 Line elemental distribution for the marked line in the SEM image for: 25% vol. Ni/Al_2O_3 composite milled for 180 minutes.

As a conclusion for the linear EDX analyses, we can affirm that the alumina particles present a nickel layer around them which leads to the idea that alumina is embedded in the nickel matrix in a almost regular manner.

The X-ray diffraction patterns of Al_2O_3/Ni powders for the starting sample and the samples milled for 120 and 180 minutes for 25% vol. Ni, are shown in Fig. 5.

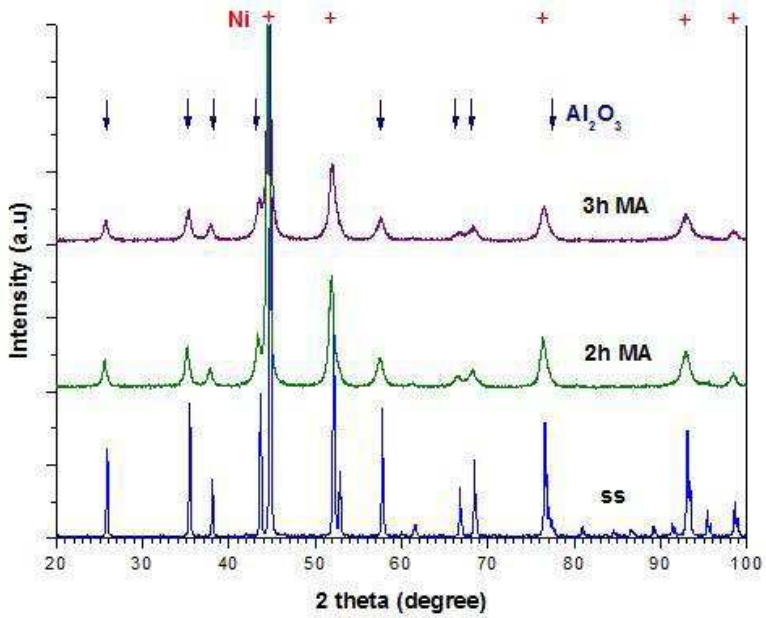


Fig. 5 X-ray diffraction patterns of Ni/Al_2O_3 composite powders obtained after 120 and 180 minutes of milling. ss denotes the starting mixture.

Only the Al_2O_3 and Ni Bragg peaks are detected in the XRD patterns for all milling times. There are no new maxima appearing in the diffraction patterns for the milled samples, so there is no evidence that a new phase was formed during the milling process (in the sensitivity limit of the X-ray diffraction). The shape of the peaks change with the milling time, the intensity is reduced and the full width at half maximum increases, suggesting that the composite contains a high amount of stresses and the crystallite mean grain size is reduced.

Using the Williams-Hall method for both nickel and alumina particle phases, it was found that after 180 minutes of milling, the grain sizes of alumina are reduced to 5.5 nm and that of nickel are 92 nm. The drastically reduction of nickel grain size can also be demonstrated by the thin layer of nickel that is seen to cover the alumina particles in the SEM recorded images. In the light of these results the obtained composite is nanometric.

Summary

Ni/Al_2O_3 nanocomposite powder, with 25% vol. Ni, was obtained by mechanical milling up to 180 minutes of milling. The formation of the nanocomposite is accompanied by particle size changes.

The images obtained by SEM analyses show a high level of homogenization of the Ni and Al_2O_3 phases when the milling times were larger than 120 minutes.

The XRD analyses highlighted only the presence of the Al_2O_3 and Ni Bragg peaks. There are no new maxima identified in the diffraction patterns for the milled samples, so there are no evidences of formation of a new phase during milling process. The grain sizes of both phases are in the nanometric range, indicating that the obtained composite has nanocrystalline structure.

Taking into account the scanning electronic microscopy, together with the X-ray diffraction analysis and EDX, it can be concluded that after 180 minutes of mechanical milling the nanocomposite Ni/ Al_2O_3 is formed.

Acknowledgements

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