Synthesis and Characterization of Binary Ni$_{75}$-Co$_{25}$ Alloy by Mechanical Alloying

Hewa.MK.Frage, Ismail.k.Jassim, Jawdat.A.Yagoob
Department of Physics, College Education of pure science, Tikrit University, Iraq

Abstract
This research work aims to investigate the binary (Ni$_{75}$Co$_{25}$) alloy prepared by mechanical alloying method, as function of milling times of (0, 2, 4, 6, 8 and 10) hr. The changes in structural and morphological properties during mechanical were investigated by several techniques respectively by X-ray diffraction (XRD) Scanning electron microscopy (SEM), and Elements dispersive spectroscopy (EDS). XRD analysis suggested several Phases formed successfully after initial milling process. Two face-centered Cubic (FCC) and hexagonal close packed (HCP) were observed prospering. The Particle size for various milling times decreased significantly with increasing time of milling. The resulted morphology shows, the milled powder a reduction of particle size which is in identical with the (XRD) Patterns. (EDS) result shows clearly atypical spectrum of Both (Ni) and (Co) only.

Keywords: Nanostructured materials, Mechanical alloying, Structural Properties, Ni-Co alloys.

1-Introduction:
A metal Matrix of (3d) transitions materials composite like Cobalt with Nickel are a topic of increasing research interest due to their versatile applications in microwave absorbers, magnetic recording devices, magnetic Sensors and different components in electronic industries [1,2]. The development of nanoparticles (NPs) based on (Ni) and (Co) have received much recent attention because of their novel catalytic, structural, optical and Magnetic properties [3,4]. Recently, Various attempts to synthesize structural and magnetic (NPs) have been reported including ball milling Chemical reduction, thermal decomposition, chemical vapor deposition and solution phase reduction [5,6]. Mechanical alloying (MA) is typically used commercially and is capable of synthesizing Materials with alloying phases in both equilibrium and non-equilibrium Manners [7]. MA is a solid-state powder Processing technique that enables production of homogenous materials starting from blended elemental Powders mixtures in the high energy ball mill [8]. The advantageous of (MA) includes the refinement changes of the grain size down to nanometer range, disordering of ordered intermetallic and possibility of alloying of difficult alloy elements [9,10]. In this research work, structural of Nano crystalline (Ni$_{75}$-Co$_{25}$) alloy Powders were prepared at various milling times by mechanical alloying process, and characterized by several techniques.

2-Experimental Procedures:
Fluke elemental Cobalt and Nickel of (99.90%) purity and particle sizes smaller than (50 µm) were separately weighted and mixed to get the desired composition of (Ni$_{75}$-Co$_{25}$). The mechni- cal alloying process was performed using a planetary high energy ball mill (Home Made). The milling process was performed at room temperature using hardened (40 balls, diameter 10mm). The ball to powder weight ratio was set a (40:1) gm. The variable milling times were used (2, 4, 6, 8 and 10)hr. The rotation speed was (137rpm). Structural and phases change in the milled powders were characterized by (XRD) using (Cu-κα) radiation (λ= 0.15406 nm). Morphology was observed via scanning electron microscopy (SEM) Model Philips (XL 30) microanalysis coupled to an energy dispersive analyzer (EDS). The variable milling times were used (0, 2, 4, 6, 8 and 10) hr.

3-Results and discussion:
Fig. 1(a-b-c) shows the x-ray diffraction Patterns for the Powders (Ni) and (Co) before milling into the (Ni$_{75}$-Co$_{25}$) alloy. It can be seen that the diffraction peaks associated with (Ni) and (Co) are typical pure spectrums with presence of peaks of (fcc) centered cubic for (Ni) and (Co) respectively [Fig.1 (a-b)]. Also hexagonal close packed Cobalt (hcp-Co) peaks dominated very clearly. No any other elements was observed as impurity for both patterns before mixing (Fig.1-c) shows the XRD pattern of mixture before milling (0hr). One can see the main diffraction Peaks of the element fcc Ni, fcc Co and (hcp) phases. The multiple main peaks could be indexed to several plans (111), (200), (220), (311) and (222) of (fcc) phases and (100), (102) of (hap) phases. The results also show that both the fcc Ni and fcc Co have nearly the same lattice parameters, which are of about (0.3531 nm) and (0.3545 nm) respectively. Fig.2 show the result of (XRD) at (2, 4, 6, 8 and 10hr) milling time. At (2hr) of milling, the same peaks are observed as in the un-milled mixture (0hr) with a high intensity especially for fcc (Ni, Co) phases with a small increase in intensity for (100) and (102) Peaks of hcp Co phase.

For (4hr) of milling, the peaks present decrease in the in the intensity. The can be attributed to several reasons such as reduction...
in particle size and the structure begins to reverse allotropic phase transformation of (Co) form hcp to fcc [11, 12]: At (6hr), Cleary shows the decreasing
Intensity of the hcc peaks until absent, but the fcc peaks of Co and Ni are still dominated and the plane (111) is only strongly dependent. Actually, the absence of solute peaks in the (XRD) pattern (8 hr) shows only phase form of fcc (Ni, Co) have been detected. For longer milling times (10 hr), a significant reduction of diffraction Peaks intensity is observed due to the severe plastic deformation [13, 14]. The results also show that the width of peaks became broadened. This is may be due to the decrement of particles size and increment of internal strain during the high energy ball milling process [15, 16].

The results also detected no any slight shift of the diffraction peaks two wards smaller (2θ) angles which means that no any lattice distortion [17]. For longer milling times (10 hr), a significant reduction of diffraction peaks intensity is also observed. This case suggests by some studies due to the stage of (l0hr) leads to finer particles. That is unsuitable for structural and magnetic properties [18].

**Fig. 1 (a, b, c): X-ray diffraction of:**

- **a-** Pure Ni
- **b-** Pure Co
- **c-** Mixing (Nis-Co25) Alloy (0hr).
Fig. 2: X-ray diffraction patterns of (Ni75-Co25). MA powders as function of total milling time, (2, 4, 6, 8 and 10) hr.

Finally, one an important can noted a decrease rapidly of crystallite size and an increase of the microstrain during the beginning of increasing milling times as shown in Fig.3. This is may be due to the pure crystalline Ni and Co, which causes no any lattice deformation of a gradual increase of internal strains with increasing milling hours [19].
Fig. 3. Average crystallite size (nm) and mean internal strain (%) of (Ni$_{75}$-Co$_{25}$) alloy versus milling times.

Fig. 4(a, b, c, d, e) shows the SEM images of the synthesized (Ni$_{75}$-Co$_{25}$) alloy at various milling times of (2, 4, 6, 8 and 10) hr. It is clear that at the case Fig.(4-a) after (2hr) of milling, both of elements Ni and Co show that approximately spherical shapes particle of uniform size. This figure also observes the existences of Ni particles are large ones and Co Particles are Clearly smaller ones. As a result of intensive fracture and cold welding as presented in Fig. (4-b). The composite (Ni$_{75}$-Co$_{25}$) particles are formed after 4 hr of milling time. For 6hr of milling time, the particles change into a platelet or a flake Shape Fig. (4-c). With milling time 8hr, one can see that there is a stage where the formation of flake shape Particles is favored, as shown for 8hr of milling time Fig. (4-d). It shows also that the plated shapes particles dominate for 8 hr of milling see Fig. (4-d). By increasing the milling time for 10 hr of milling, the mechanical alloying progress and the refinement of particles size continues [18]. The longest milling time 10 hr Fig. (4-e) is clearly shown that the majority of particles grains exhibit around shape with small diameter, however one can still note the presence of some big ones having a platelet shape. [19].
Fig. 4. (a-b-c-d-e) SEM micrographs of Ni$_{75}$Co$_{25}$ for various milling time:

\[ a = 2\text{hr}, \ b= 4\text{hr}, \ c = 6\text{hr}, \ d = 8\text{hr} \text{ and } e = 10\text{hr}. \]

The elemental mapping by EDS Coupled to the SEM shows the spectroscopy of Ni and Co distribution for the selected areas of some samples for 10 hr of milling (Fig. 5). It is clearly shown the Nickel and Cobalt are still separated from each other and it the beginning becomes difficult to distinguish Ni and Co particles, this indicated that Ni and Co are in complete alloying process indicating that two elements are completely alloyed and the (Ni-Co) solid solution is formed [20]. Actually these results of EDS are identical and consistent with XRD analysis. Also, we noted that no any element or (O$_2$) contamination from the milling media was observed for all milling times.

Fig. 5. Typical EDS spectrum of Ni$_{75}$Co$_{25}$ at 10hr milling time.

4-Conclusion:

The effect of mechanical alloying on the structural and morphological properties of (Ni$_{75}$Co$_{25}$) alloy prepared from pure elemental Ni and Co Powders has been investigated the structural characterization of the alloy at various milling times of (2, 4, 6, 8, and 10) hr were studied by XRD, SEM and EDS measurements. XRD analysis indicates the formation of two main solid solution Ni (Co) and Co (Ni) with a face centered structure. The allotropically Structural transformation of Co from (hcp) to (fcc) has been identified. The hcc-Co peaks completely disappear with increasing time of milling. The variations of the Particle size results are decreased significantly with increasing milling times. The SEM images taken at different milling time allowed us to follow the morphology of the (Ni$_{75}$Co$_{25}$) alloy at different stages. The results of EDS show no any strange element or oxidation contamination from the milling media.
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